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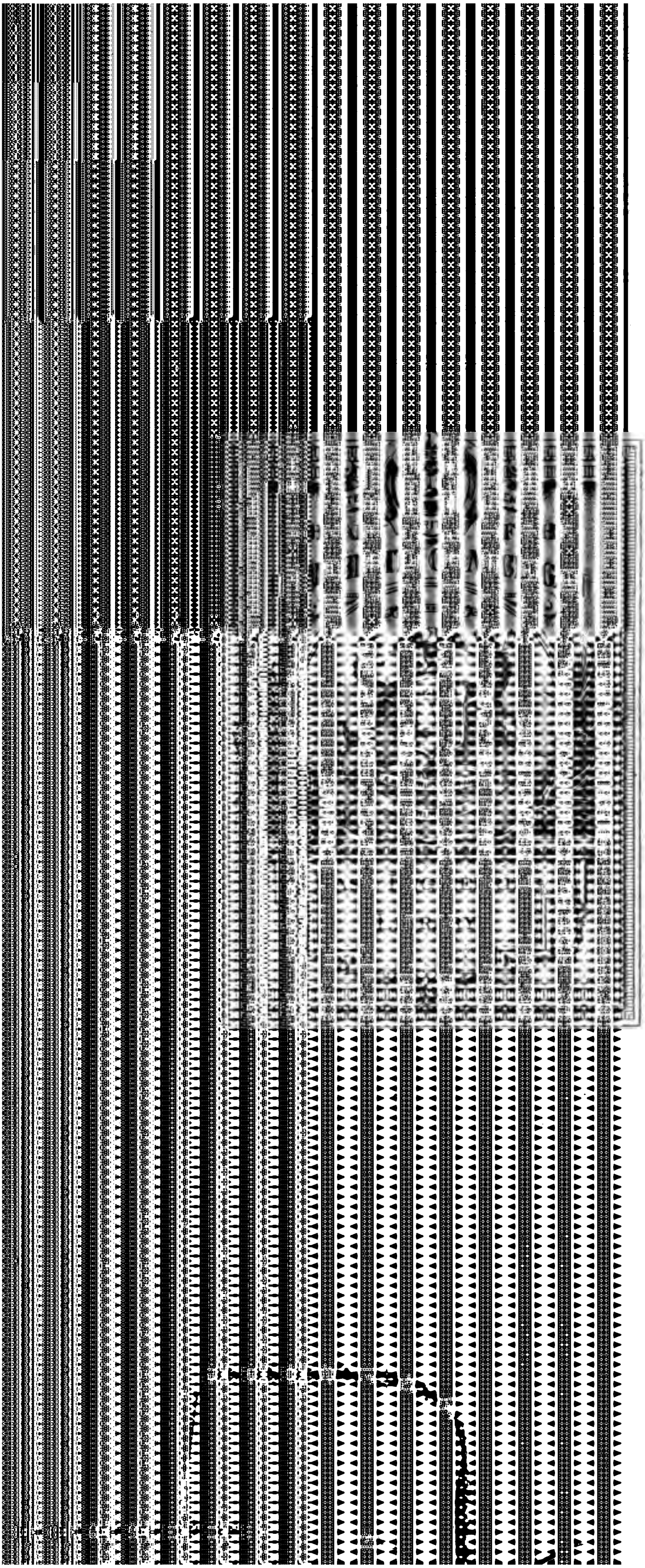

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DRYING OILS, BOILED OIL, AND SOLID AND LIQUID DRIERS

DRYING OILS, BOILED OIL, AND SOLID AND LIQUID DRIERS

A PRACTICAL WORK FOR MANUFACTURERS OF OILS,
VARNISHES, PRINTING INKS, OIL-CLOTH AND
LINOLEUM, OIL-CAKES, PAINTS, ETC.

BY

LOUIS EDGAR ANDÉS

AUTHOR OF
"ANIMAL FATS AND OILS" "VEGETABLE FATS AND OILS"
"IRON-CORROSION, ANTI-FOULING, AND ANTI-CORROSIVE PAINTS" ETC.

WITH FORTY-TWO ILLUSTRATIONS

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PREFACE

After more than thirty-five years' practical experience of the manufacture of varnishes, boiled oil, paint, and printing ink, having followed the progress of this industry and also the scientific achievements in the domain of the drying oils, I may, without great presumption, consider myself qualified to treat of the drying oils, their preparation, and investigation.

My present work contains detailed information as to the source, extraction, and properties of all oils of any technical value, as to their treatment for the most varied uses—for boiled oil, paints, printing ink, oilcloth, and linoleum—my extensive experience in these matters, and the portion of chemistry necessary for a general comprehension. Specially detailed sections are devoted to the preparation of the liquid and solid siccatives, driers, and substitutes for boiled oil; the concluding portions deal with the examination of the drying oils and boiled oils for adulteration and suitability for practical purposes. I hope that this work, which is intended to represent the present state of the knowledge of the subject, may meet with friendly reception in interested circles.

LOUIS EDGAR ANDÉS.

Vienna, *February* 1901.

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DRYING OILS, BOILED OIL, SOLID AND LIQUID DRIERS

CHAPTER I

GENERAL CHEMICAL AND PHYSICAL PROPERTIES OF THE DRYING OILS—CAUSE OF THE DRYING PROPERTY—ABSORPTION OF OXYGEN—BEHAVIOUR TOWARDS METALLIC OXIDES, ETC.

THE drying oils, like all vegetable fats and oils, are contained in the seeds and fruits; they are amongst the most widely distributed vegetable products. In almost all the tissues of phanerogams and cryptogams fats and oils occur in small quantity, generally in the contents of the cells, and rarely in the cell wall. The oil generally remains in the tissue where it is formed, and becomes enclosed there; only rarely does it break forth in some unknown manner from the place where it is produced and cover the neighbouring organs by a more or less thick layer. In small quantity, oil or fat occurs in the protoplasm of young cells; in larger amount, it is found as a constituent of the watery cell sap in the form of small drops or small spheres together with protoplasm residues, which are also suspended as granular or slimy masses in the cell contents. Accompanying the oil or fat in the cells are starch granules, aleurone grains, chlorophyll, resinous colouring matters, resin granules, colouring matters dissolved in the oil, etc. The drops or spheres of fat are as a rule suspended in the cell sap; in certain cases, *e.g.* in the pulp of the olive, which yields the non-drying olive oil, the drops of oil appear to be surrounded by a peculiar delicate membrane ("fat-bladder"). According to the investigations of Sachs, the aleurone grains so frequently found in oil-bearing seeds are aggregations of fat and albumin particles. As regards the formation of fats and oils in the plant cells, almost nothing is known; whether the oil or fat is immediately formed from particles of the protoplasm, or arises from spherical globules suspended in the cell sap, is not yet known. (From the seeds and fruits the drying oils can be obtained by hot or cold pressing, or by extraction by readily volatile solvents such as benzine, carbon bisulphide, etc.)

All the drying oils are fluid at the ordinary temperature, and in different measure for many degrees below 0° C. Linseed oil is one of the most fluid; it does not solidify at the lowest temperatures of our latitudes. The boiling points are at different high temperatures; the majority of the oils decompose at the same time. The colours of the oils vary from pale yellow, bright yellow, and dark yellow to green, the characteristic colour of hempseed oil. The drying oils differ also in odour and taste; many of them, when obtained by cold pressing, are used as edible oils. (Like all fats and oils, the drying oils are insoluble in water, with which they form emulsions in the presence of small quantities of alkalis, by larger quantities of which they are saponified. With the exception of castor oil, they dissolve only in small quantity in cold alcohol; but, on the contrary, are soluble in hot or cold ether, benzene, petroleum ether, carbon bisulphide, chloroform, and turpentine.) All the drying oils produce grease spots upon paper; they burn from a wick with a more or less luminous flame, producing larger or smaller quantities of soot. They dissolve sulphur and phosphorus, and unite with chlorine and sulphur chloride to form caoutchouc-like substances. Sulphuric and nitric acids react violently with the drying oils.

The drying oils, like all vegetable oils, consist of the neutral glycerin esters (glycerides) of the fatty acids; they thus possess the general formula $C_3H_5(OCOR)_3$, in which R represents a monovalent carbon radical. They also contain free fatty acids in larger or smaller quantity.

Maumené, and later, Fehling, observed that the rise of temperature produced when fatty oils are mixed with sulphuric acid is different for the different oils, and that the greatest rise occurs with the drying oils. This increase is with

Olive oil	42° C.
Sesamé oil	68° C.
Castor oil	47° C.
Poppy oil	74°·5 C.
Hempseed oil	98° C.
Walnut oil	101° C.
Linseed oil	132° C.

In order to obtain comparable results it is necessary always to work under exactly the same conditions, for the rise of temperature depends not only upon the nature of the particular oil, but also upon numerous other conditions; thus, upon the quantity and strength of the acid, the temperatures of the two liquids, the duration of the operation of mixing, the nature of the vessel, etc. When the temperatures of the oil and acid have been noted, both are weighed off into small glass vessels and mixed whilst stirring with a delicate thermometer; the highest temperature shown by the thermometer is observed. A peculiar sign of a drying oil, as opposed to a non-drying oil, is afforded by the "elaïdin test," in which the oleïn of the oils is converted into elaïdin by the action of nitrous acid. The non-drying oils become solid or buttery, the drying oils on the contrary become pasty or remain unaltered; this property affords a method for testing the purity of an oil, by observations of the time required for solidification and the colour of the solidified mass. Linseed, poppy, and walnut oils remain unaltered; cottonseed, sunflower, and beech-nut oils become pasty. The elaïdin test is carried out by mixing in a large test-tube, with continued violent shaking, 10 volumes of oil, 1 volume of water, and 2 volumes of fuming nitric acid of specific gravity 1·45. After some time a saturated solution of potassium nitrite is carefully allowed to run down into the test-tube, the time and manner of the solidification are then observed.

Mulder makes the following statements as to the composition of linseed oil: it is a mixture of linoleïn, as principal constituent, with oleïn, palmitin, and myristin; it has the following percentage composition:—

	A.	B.
C	76·9	76·8
H	11·1	11·2
O	12·0	12·0

According to Mulder, poppy oil has the same composition as linseed oil; its percentage composition is

	A.	B.	C.
C	76·5–76·7	77·2	76·5
H	11·5–11·8	11·4	11·2
O	11·8	11·4	12·2

Its principal constituent is linoleïn; it contains also oleïn, myristin, and laurin.

Lefort gives the following as the composition of hempseed and walnut oils:—

	Hempseed oil.		Walnut oil.	
C	71·0	70·9	70·6	70·6
H	11·8	11·8	11·6	11·7
O	17·2	17·3	17·8	17·7

whilst Mulder found

	Hempseed oil.	Walnut oil.	
C	76·0	76·1	76·0
H	11·3	11·3	11·2
O	12·7	12·6	12·8

Walnut oil contains oleïn, myristin, much laurin, also linoleïn; it does not contain stearin or palmitin.

From his investigations of linseed, poppy, and walnut oils Mulder draws the following conclusions:—

1. The drying oils which were examined differ in percentage composition, and thus cannot be regarded as the same mixture of the same fats, although they may contain the same drying principles. Also they cannot have the same composition as non-drying oils.

2. Stearin was not found in any of the drying oils examined. Linseed oil contains a certain quantity of palmitin and myristin; poppy and walnut oils contain myristin and laurin. Each of the oils mentioned contains a certain amount of oleïn. The percentage composition of the oils accords with these constituents.

3. There are no grounds for recognising any differences in a pure drying oil of the same species, age, etc., which are of importance in view of its use by artists and painters.

Mulder called the principal constituent of linseed, poppy, walnut, and apparently of all drying oils, linoleïn, $C_6H_5O_3(C_{32}H_{27}O_3)_3$,[1] the important component of which is an acid, linoleïc acid, $HO.C_{32}H_{27}O_3$, which is set free by saponification.

The substances produced from linseed oil in drying, according to Mulder, are

Linoleïc anhydride, $C_{32}H_{27}O_3$, a slimy mass like melted indiarubber.

Linoxic acid, $HO.C_{32}H_{25}O_9$, obtained by the oxidation of linoleïc acid in air, either of the free acid or of its compound with lead oxide. There are two forms of this acid, one colourless, the other red; each can be changed into the other. Heat turns the white acid red, light turns the red acid white. Alkalis change the white to red, as also strong acids, by which the amount of combined water is decreased by one equivalent.

---

[1] Old notation, C=6, O=8.

Linoxyn, $C_{32}H_{27}O_{11}$, is a product of the oxidation in air of linseed, poppy, and walnut oils. Bases transform it into linoxic acid. By heat it is turned red, but again becomes white on exposure to light. Thus there are both white and red modifications of linoxyn. Its constitution is expressed by the formula $2HO.C_{32}H_{25}O_9$, which should be compared with that of linoxic acid. Mulder states that oleïn or oleïc acid is a constituent of the drying oils; it produces sebacic acid on distillation, which is not accompanied by linoleïn or linoleïc acid.

Since the important researches of Mulder, which were carried out at a tolerably early period (the German translation of the Dutch original appeared in 1867), the work of Hazura and Bauer has somewhat altered our knowledge of the subject. The results are given below, so far as they are necessary to an understanding of the composition of the drying oils.

Bauer and Hazura found in hempseed oil as the principal constituent hempolic acid, which on fusion with caustic potash at a low temperature produces chiefly myristic, acetic, and formic acids; azelaïc acid is also obtained in small quantity. From 100 parts of hempolic acid 55 parts of crude fatty acids were obtained, which were regarded as myristic acid. The chief product of the oxidation of hempolic acid is sativic acid, which forms salts with metals.

Hempolic is an acid of the series $C_nH_{2n-4}O_2$, and according to analytical results has the same composition as linolic acid, $C_{18}H_{32}O_2$.

Sunflower oil contains principally the glycerides of linolic acid, $C_{18}H_{32}O_2$, and oleïc acid, $C_{18}H_{34}O_2$. Since the latter is present in but small amount, the liquid fatty acids of sunflower oil form the most suitable material for a study of linolic acid. These acids would be obtained by saponifying the oil with alcoholic potash, dissolving in water, neutralising by acetic acid, precipitating with lead acetate, extracting the lead salts by ether, and decomposing the ethereal solution by dilute sulphuric acid.

In the course of their investigations Hazura and Bauer found that linoleïc acid is not a single acid, but contains four different fatty acids. It consists of 80 per cent. of linolenic acid, $C_{18}H_{30}O_2$, with isolinolenic acid, $C_{18}H_{30}O_2$, linolic acid, $C_{18}H_{32}O_2$, and oleïc acid, $C_{18}H_{34}O_2$. Dilute alkaline solutions of the liquid fatty acids of linseed oil give, on oxidation by potassium permanganate, dihydroxystearic acid, $C_{18}H_{34}O_2(OH)_2$, tetrahydroxystearic (sativic) acid, $C_{18}H_{32}O_2(OH)_4$, linusic acid, $C_{18}H_{30}O_2(OH)_6$, and isolinusic acid, $C_{18}H_{30}O_2(OH)_6$, also small quantities of azelaïc acid, $C_9H_{16}O_4$, and other lower fatty acids. In the oxidation of concentrated alkaline solutions, through breaking up of the molecules, azelaïc acid is formed as the principal product.

In the bromination of the fatty acids of linseed oil are formed oleïc acid dibromide, linolic acid tetrabromide, linolenic acid hexabromide, and isolinolenic acid hexabromide. The liquid fatty acids of hempseed, poppy, walnut, and cottonseed oils are not individual acids, but mixtures of unsaturated fatty acids.

The liquid fatty acids of hempseed oil consist chiefly of linolic acid, $C_{18}H_{32}O_2$, with small quantities of linolenic acid, $C_{18}H_{30}O_2$, isolinolenic acid, $C_{18}H_{30}O_2$, and oleïc acid, $C_{18}H_{34}O_2$.

The liquid fatty acids of walnut oil consist principally of linolic, linolenic, isolinolenic, and oleïc acids.

The liquid fatty acids of poppy oil contain linolic acid as principal constituent, considerable quantities of oleïc acid, and very small amounts of linolenic and isolinolenic acids.

The liquid fatty acids of cottonseed oil contain no acid of the series $C_nH_{2n-6}O_2$; they consist of two parts of linolic acid to one part of oleïc acid.

According to Hazura, the acids of the drying oils all behave towards the oxygen of the air in a similar manner. The rapidity of the oxidation depends on the proportion of linolic to linolenic acid in the particular oil. The more linolenic acid is present, the more rapid is the oxidation. The oxidation, however, does not consist alone in the saturation of the free valencies with oxygen, but oxygen is interposed between carbon and hydrogen, so that the products contain alcoholic hydroxyl groups. There is no difference between the oxidation of the acids of the drying oils and of their salts. When thin layers of these acids are exposed to the air at the ordinary temperature, or when the temperature at which the oxidation takes place is about 80° C., anhydrides are formed at the conclusion of the oxidation. From the slimy resinous products of oxidation solid substances are finally formed, which are insoluble in ether, but are again converted into acids soluble in ether when heated with alkalis. It is impossible to decide in what manner these anhydrides are formed; whether from two molecules of the hydroxy-acids one molecule of the anhydride is formed, or whether an inner anhydride is produced. In this connection it is to be noted that, in all processes in which oils dry, only linolic, linolenic, and isolinolenic acids participate, since it is known that oleïc acid forms no solid product by oxidation with the oxygen of the air.

Hazura doubts the existence of Mulder's red linoxic acid. Mulder is incorrect in stating that all the glycerin of linseed oil is first oxidised and that the oxidation commences with the glycerin of the glyceride of linoleïc acid. Old samples of linseed oil have been found to have an acid value of 8·9 and 12·2; this shows that oxidation may commence with the glycerin, but very probably only with that contained in oleïn, palmitin, and myristin. Accordingly linolin is oxidised to the glyceride of hydroxylinolic acid, and there is produced a substance soluble in ether, which should be called hydroxylinolin, in order to distinguish it from Mulder's linoxyn. It cannot be ascertained what happens next, whether by the longer action of the air this substance is converted into the anhydride of hydroxylinolic acid (Mulder's linoxyn), neither can we say what alterations the free acids, oleïc, palmitic, and myristic, undergo.

X The most important property of the drying oils is that, when exposed in thin layers to air, light, and heat, they dry to a solid, elastic skin, and in thick layers or in bulk thicken so that they become sticky and almost syrupy. By the action of air alone drying is slow; it is more rapid when light assists, and most rapid under the influence of heat; it takes place with the greatest ease when certain substances are added to the oil which facilitate the drying. It was formerly believed that additions of certain substances, such as metallic compounds rich in oxygen, assisted drying by giving up oxygen to the oils at an elevated temperature, so that the fatty acids of the oils were oxidised to a certain extent, and that the oxidation thus initiated could then proceed to the point of solidification. This opinion was accepted for many years; upon it were based the different methods for preparing boiled oil. However, an observation of Chevreul should have been sufficient to indicate the true theory of oil boiling; he found that the oxygen compounds of lead and manganese simply absorb oxygen from the air and transfer it to the oil. When linseed oil, which has been treated with manganese, is mixed with raw linseed oil, there results a product which absorbs oxygen far more rapidly than if each constituent were taken alone.

Thus there is no doubt that the quantity of oxygen absorbed in a fixed period of time does not proceed from the manganese oxide in the oil, because it contains less oxygen than the manganese oil. The explanation is as follows. The raw oil absorbs in the same time a larger quantity of oxygen, because it is offered to the oil under the most favourable conditions; the oxygen is taken up by the manganese oxide, and by it transferred to the oil; in this manner a continuous conveyance of the oxygen of the air to the manganese oxide, and thence to the oxidisable constituents of the oil, takes place.

From his researches on the behaviour of the several constituents of linseed oil in drying, alone and in combination with metallic oxides, Mulder drew the following conclusions:—

1. At the ordinary temperature the oxidation of linoleïc acid, when spread out in a very thin layer, attains a maximum in several days.

2. Linoleïc acid, in combination with lead oxide, is completely oxidised to linoxyn by contact with air; the product is resinous in the free state, but by long contact with lead oxide becomes hard and brittle.

3. In the drying of linseed oil two periods are to be distinguished—(*a*) to the formation of linoxyn (the oil dries, but remains elastic); (*b*) the transformation of linoxyn into a compound containing more oxygen (the layer becomes brittle, and loses its durability).

4. Linoxyn is the real basis of the durability of all paints and varnishes; it remains long unaltered and elastic when little exposed to sunlight; the oxidation of linoleïn in semi-darkness is considerably slower than in the light, but otherwise is quite the same.

5. The opinion that linseed oil, which has simply been heated to 70° to 100° C., dries better than ordinary oil, is completely erroneous.

6. Strong sulphuric acid makes linseed oil more capable of absorbing oxygen.

7. Manganese borate is effective because it absorbs oxygen and readily gives it up again to the oil.

8. The action of litharge is different according as it is heated with linseed oil or only mixed with it.

9. Linseed oil mechanically refined by treatment with animal charcoal does not dry more quickly than the raw oil; thus raw linseed oil contains no substance which hinders drying.

10. Red lead and other substances, which give off oxygen, cause the formation of linoxic acid; the heavy metallic oxides only cause the production of linoxic acid compounds when they are heated with the linseed oil and exposed to the air.

11. The drying properties of linseed oil which has been boiled with litharge cannot be increased by simple admixture with metallic oxides rich in oxygen.

On examining these conclusions two points are especially evident, which show that Mulder recognised the different action of different driers. In (7) he says: " Manganese borate is effective because it absorbs oxygen and readily gives it up again to the oil," and he is of the opinion that this substance causes a peculiar chemical action on the oil. In (8), on the other hand, " the action of litharge " occurs, and in the arguments upon which the conclusions are based the following statements are made. The actions of litharge and red lead are founded on different principles, both do not act in the same manner; the latter can give up oxygen, the former acts simply as lead oxide. Red lead paints are known to be very hard; in fact, red lead gives off oxygen, and lead linoxate is produced. At the ordinary temperature litharge has not the property of hardening linseed oil; red lead thus acts partially as lead oxide and partially as an oxidising agent. Litharge differs in its action according as it is boiled with linseed oil or simply mixed with it. Red lead and other oxidising substances cause the formation of linoxic acid; all other heavy metal oxides produce linoxates only when they are boiled or heated with linseed oil exposed to the air.

Mulder thus recognises two different processes as the cause of the drying of oils—(1) the absorption of oxygen by substances (manganese borate) contained in the oil and its transference to the oil, and (2) the actual absorption of oxygen by the oil from added metallic oxides rich in oxygen.

The increase of weight in drying, owing to absorption of oxygen from the air, was proved by Mulder by numerous experiments. More recently Weger and Lippert have taken up the subject; an account of their results follows. Weger made a series of determinations of the oxygen absorbed by linseed oil and by oil boiled with different driers. The oils were spread out in the thinnest possible layer upon glass plates of about 88 sq. mm. surface and weighing about 25 grms.; it is rather difficult properly to spread out thickened oils. These tests do not give absolute valid results, since the rapidity of the drying depends on the temperature, light, air, etc.; the process for determining the absorbed oxygen can only be used to obtain relative figures when parallel experiments are undertaken at the same time; a mean value is obtained from the results of a large number of experiments. The same holds good for the determination of the time of drying. Weger generally considered the oils to be dry when the skin of oil did not adhere to the finger lightly pressed on. In the case of raw and boiled linseed oil the maximum weight is obtained when the oil is just dry, or in twelve to twenty-four hours after drying. The time at which the oils are hard, *i.e.* when the layer can bear a strong pressure with the finger, must in every case be regarded as coinciding with the occurrence of the maximum weight.

Weger observed neither with raw nor boiled linseed oil, after the layers were hard, any considerable increase of weight, *i.e.* none apart from that due to atmospheric conditions. Still, the probability of a further absorption of oxygen cannot be denied, but this must be smaller than the loss of volatile materials. The thickened oils, boiled rosin oil and wood oil, give large deviations between the time of drying and of attaining the maximum weight. All the linseed oils used by Weger in these experiments were genuine. When the drying period and the increase of weight are graphically represented, there is found to be a slow, then a rapid, rise of the curve, slow increase to a maximum, longer or shorter duration at this height, tolerably rapid descent, and finally quite a slow decrease.

[TABLES.

ABSORPTION OF OXYGEN (INCREASE OF WEIGHT) OF DIFFERENT RAW LINSEED OILS WITHOUT DRIERS. (MARKED * WHEN DRY.)

	Linseed Oil from East Indian Seed.	Artists' Linse O .	English Linseed Oil, kept 5 years in closed glass bottle.	Linseed Oil from East Indian Seed, heated to 150° C.	Linseed Oil from East Indian Seed, blown in the cold.	Linseed Oil from East Indian Seed, blown 25 hrs. cold and 25 hrs. hot.	Artists' Linseed Oil, heated to 280° C.	Linseed Oil, heated to 250° C. by introduction of superheated Steam.	Linseed Oil, blown at 100°-140° C., kept 3 years.	Stand Oil.
Number of experiment .	1	4	7	11	12	14	15	17	19	21
Weight of oil, decimgrms.	295	318	294	382	323	450	503	341	512	530
Time at which exposed .	Summer 6 p.m.	Summer 10 a.m.	Summer 5 p.m.	Summer 11 a.m.	Summer 5 p.m.	Summer 4.30 p.m.	Summer 5 p.m.	Summer 6 p.m.	Summer 4 p.m.	Summer 6.30 p.m.
Increase of weight after										
20 hours .	..	..	..	..	..	..	..	..	..	-1 = -0·2%
1 day .	..	..	..	..	..	..	..	..	46 = 9%	..
1½ ,, .	9 = 3%	..	8 = 2·7%	..	0 = 0%	9 = 2·1%	7 = 2·1%	4 = 1·1%	..	6 = 1·1%
2 days .	..	1 = 0·3%	..	2 = 0·5%	..	..	..	..	77 = 15·1%*	..
2½ ,, .	26 = 8·8%	..	31 = 10·5%	..	0 = 0%	21 = 5·1%	15 = 4·4%	..	..	21 = 4·0%
3 ,, .	..	..	..	..	..	..	..	17 = 4·1%	73 = 14·3%	..
3½ ,, .	47 = 15·9%	..	58 = 19·7%*	..	7 = 2·2%	31 = 7·5%	23 = 6·8%	..	73 = 14·3%	26 = 4·9%
4 ,, .	..	12 = 3·8%	..	36 = 9·9%	..	32 = 7·7%	..	44 = 12·9%	..	..
4½ ,, .	..	..	..	..	17 = 5·3%	31 = 7·5%	45 = 13·2%	..	70 = 13·7%	36 = 6·8%
5 ,, .	50 = 16·9%	24 = 7·5%	53 = 18·1%	57 = 14·9%	..	..	..	53 = 15·5%*	..	..
5½ ,, .	..	..	..	..	37 = 11·5%	34 = 8·2%*	56 = 16·5%*	..	..	40 = 7·5%
6 ,, .	51 = 17·3%*	24 = 7·5%	52 = 18·0%	65 = 17%*	..	..	..	51 = 15·0%	..	..
6½ ,, .	48 = 16·3%	..	..	..	48 = 14·9%	32 = 7·7%	..	53 = 15·5%	..	47 = 8·9%
7 ,, .	..	50 = 15·7%	..	63 = 16·5%	..	..	57 = 14·5%	..	..	..
8 ,, .	..	54 = 17·0%*	..	58 = 15·2%	54 = 16·7%*	..	51 = 13·0%	..	..	51 = 9·6%
9 ,, .	42 = 14·2%	52 = 16·7%	49 = 16·7%	58 = 15·2%	..	..	..	50 = 14·7%	..	..
11 ,, .	..	47 = 14·8%	..	54 = 14·1%	49 = 15·2%	..	..	..	..	49 = 9·2%
13 ,, .	..	44 = 13·8%	..	..	..	..	..	..	..	51 = 9·6%
14 ,, .	..	43 = 13·5%	..	..	47 = 14·6%	..	..	..	..	54 = 10·2%
18 ,, .	38 = 12·9%	..	41 = 13·9%	..	..	..	..	44 = 12·9%	..	59 = 11·1%*
27 ,, .	..	42 = 13·2%	..	50 = 13·1%	..	..	..	..	63 = 12·3%	57 = 10·8%
30 ,, .	..	..	..	..	38 = 11·8%	..	..	..	..	..
1 ,, .	..	..	..	..	..	33 = 7·9%	..	..	..	..
39 ,, .	..	..	39 = 13·3%	..	..	..	..	..	..	..
44 ,, .	29 = 10·0%	..	..	48 = 12·6%	..	..	..	..	..	..
53 ,, .	..	37 = 11·6%	..	..	..	..	..	..	..	52 = 9·8%

ABSORPTION OF OXYGEN (INCREASE OF WEIGHT) OF BOILED LINSEED OIL WITH DIFFERENT DRIERS.

	Commercial Oil, boiled with Litharge.		Commercial Oil, apparently made with 0·3% of Manganic hydroxide at 220° C.		Heated with 0·4% of Litharge for 3 hrs. at 200° C.	Heated with 3% of Red Lead for 3 hrs. at 200° C.	"Boiled" Oil made by means of the Electric Current.		English Ozonised Boiled Oil, with Lead and Manganese Resinates.	English Lead and Manganese Boiled Oil.	Double Boiled Oil.	Orenva Boiled Oil.
Number of experiment .	34	35	36	37	38	39	40	41	42	43	44	45
Time at which exposed .	Summer 6 p.m.	Winter 9 a.m.	Summer 6 p.m.	Winter 9 a.m.	Winter 12 noon.	Winter 12 noon.	Summer 4 p.m.	Summer 5.30 p.m.	Winter 11 a.m.	Summer 4 p.m.	Summer 5.30 p.m.	Summer 5 p.m.
Weight of oil, decimgrms.	520	406	336	551	707	523	516	275	425	447	408	278
Increase of weight after												
3 hours .	..	13= 3·2%	..	5= 0·9%	..	..	..	..	..	..	..	..
9 ,, .	..	52=12·9%	..	7= 1·3%	..	..	..	..	22= 5·0%	..	..	..
16 ,, .	76=14·6% (dry)	..	3= 0·8%	75=13·6%	..	..	35= 6·8%	17= 6·2%	..	68=15·2% (dry)	67=16·4%	39=14·0%
1 day .	74=14·6%	60=14·8% (dry)	13= 3·7%	81=14·7% (dry)	55= 7·8%	63=12·1%	70=13·5%	37=13·4%	57=12·9%	66=14·8%	67=16·4%	42=15·1%
1½ ,, .	74=14·6%	58=14·3%	48=14·3%	..	..	..	75=14·5%	46=16·7% (dry)	58=13·6%	68=15·2%	64=15·7%	44=15·8%
2 days .	..	..	52=14·8%	..	87=12·3%	67=12·8%	78=15·1% (dry)	45=16·3%	..	..	..	..
2½ ,, .	76=14·6%	..	47=13·4%	..	..	..	..	45=16·3%	..	65=14·6%	58=14·2%	42=15·1%
3 ,, .	..	58=14·3%	..	..	94=13·3% (dry)	73=14·0% (dry)	68=13·2%	..	64=14·4%	..	..	..
3½ ,, .	73=14·0%	..	44=12·5%	78=14·2%	..	..	..	41=14·9%	..	..	..	..
4 ,, .	..	..	..	..	93=13·2%	64=12·2%	..	..	..	..	..	..
5 ,, .	..	..	..	..	92=13·0%	60=11·5%	..	..	72=16·2% (dry)	..	..	..
9 ,, .	..	..	..	..	87=12·3%	60=11·5%	..	37=13·4%	72=16·2%	..	..	..
23 ,, .	..	..	..	..	..	..	..	..	..	57=12·8%	51=12·5%	..
26 ,, .	..	58=14·3%	..	69=12·5%	..	..	51= 9·9%	..	..	..	..	..
28 ,, .	65=12·5%	..	37=10·5%	..	..	..	..	..	..	..	..	..

ABSORPTION OF OXYGEN (INCREASE OF WEIGHT) BY BOILED LINSEED OILS MADE WITH RESINATES.

	Linseed Oil with 2% of Lead-Manganese Resinate, without heat.	Linseed Oil, heated to 280° C., then 2% of Lead-Manganese Resinate without heat.	Linseed Oil, blown cold with 2% of Lead-Manganese Resinate.	Linseed Oil, blown at 150° C., then cold with 2% of Lead-Manganese Resinate.	Varnish Linseed Oil, treated in cold with Manganese Resinate; the Oil contains 0·10% of Manganese.				Varnish Linseed Oil, with Manganese Resinate, then heated for a short time to 170°-180° C.		
Number of experiment .	23	24	25	26	27	28	29	30	31	32	33
Weight of Oil, decimgrms.	548	501	556	401	644	244	455	322	351	335	504
Time at which exposed .	Winter 9 a.m.	Winter 9 a.m.	Winter 9 a.m.	Winter 9.30 a.m.	Summer 6.30 p.m.	Summer 6.30 p.m.	Summer 6.30 p.m.	Summer 6.30 p.m.	Summer 5.30 p.m.	Summer 7 p.m	Summer 4.30 p.m.
Increase of weight after											
5 hours .	77=14·1%	77=15·3%	71=12·8%	58=14·5%	..	..	..	..	..	..	..
8½ ,, .	96=17·5% (dry)	87=17·4% (dry)	93=16·7%	66=16·5%	..	..	..	..	..	..	..
12 ,, .	..	..	..	..	16= 2·3%	28=11·5%	02= 4·4%	27= 8·4%	..	3= 0·9	72=14·3% (dry)
1 day .	..	87=17·4%	95=17·1%	66=16·5%	100=15·5% (dry)	39=16·0% (dry)	67=14·4% (dry)	48=14·9% (dry)	54=15·4% (dry)	46=13·7%	72=14·3%
1½ ,, .	96=17·5%	87=17·4%	98=17·6% (dry)	70=17·5% (dry)	94=14·6%	34=13·9%	64=14·1%	46=14·3%	49=14·0%	48=14·3 (dry)	67=13·3%
2 days .	..	..	..	..	.	..	..	..	..	44=13·1	..
2½ ,, .	95=17·3%	..	..	70=17·5%	..	..	..	..	..	..	..
3 ,, .	..	..	..	..	..	..	..	..	46=13·1%	..	..
5 ,, .	..	87=17·4%	..	..	..	..	..	..	42=12·2%	..	..
9 ,, .	..	87=17·4%	91=16·3%	..	..	..	..	..	..	..	..
14 ,, .	..	80=16·0%	86=15·5%	19=17·2%	..	..	..	..	..	..	..

ABSORPTION OF OXYGEN (INCREASE OF WEIGHT) BY ROSIN OIL AND BOILED ROSIN OILS.

	Rosin Oil without Driers.				Rosin Oil, 6% of Lead-Manganese Resinate dissolved at 120° C.			
Number of experiment .	62	63	64	65	66	67	68	69
Weight of Oil, decimgrms.	287	314	435	399	364	379	431	372
Time at which exposed .	Summer 6 p.m.	Summer 6 p.m.	Summer 7 p.m.	Summer 12 noon.	Summer 6 p.m.	Winter 5 p.m.	Summer 6.30 p.m.	Summer 5 p.m.
Increase of weight after								
1 day .	-9=-3·1%	-6=-1·9%	-8=-1·8%	..	31=8·5%	13=3·4%	64=14·9%	..
1½ ,, .	-13=-4·5%	..	-13=-3·0%	..	63=17·3%	..	88=20·4%	..
2 days .	..	-4=-1·2%	..	..	..	58=15·3%	..	..
2½ ,, .	-18=-6·3%	-7=-2·3%	-16=-3·7%	0=0·0%	71=19·5%	..	96=22·3%	75=20·2%
3 ,, .	..	..	..	..	..	67=17·6%	..	..
3½ ,, .	..	..	..	..	..	..	..	81=21·8%
4½ ,, .	-17=-5·9%	..	-13=-3·0%	-11=-2·8%	77=21·2%	..	103=23·9%	..
5 ,, .	..	..	..	-10=-2·6%	..	73=19·3%	..	81=21·8%
5½ ,, .	-25=-8·7%	-4=-1·2%	-16=-3·7%	..	77=21·2%	..	106=24·6%	83=22·3% (dry)
6½ ,, .	-32=-11·1%	..	-13=-3·0%	..	82=22·5%	..	111=25·7% (dry)	..
7 ,, .	..	-6=-1·9%	..	..	..	..	..	..
7½ ,, .	-34=-11·5%	..	-14=-3·2%	-10=-2·6%	85=23·3% (dry)	..	109=25·3%	..
8 ,, .	..	-12=-3·8%	..	..	..	..	..	..
8½ ,, .	-42=-13·9%	..	-17=-3·9%	..	82=22·5%	..	109=25·3%	..
9 ,, .	..	..	..	-11=-2·8%	..	75=19·8%	..	81=21·8%
10 ,, .	-42=-13·9%	-10=-3·1%	-24=-5·5%	..	..	..	108=25·0%	..
11 ,, .	..	..	..	-16=-4·0%	..	..	..	..
12 ,, .	..	-14=-4·5%	..	..	..	..	..	83=22·3%
13 ,, .	-46=-16·0%	..	-26=-6·0%	..	82=22·5%	..	107=24·8%	..
14 ,, .	..	-21=-6·7%	..	..	..	..	..	79=21·2%
15 ,, .	-52=-18·1%	..	-31=-7·1%	..	82=22·5%	..	..	..
19 ,, .	-62=-21·6%	..	-42=-9·7%	..	82=22·5%	..	108=25·1%	..
21 ,, .	-62=-21·6%	..	-42=-9·7%	..	84=23·0%	..	..	..
48 ,, .	-76=-26·5%	..	-66=-15·2%	..	79=21·7%	..	107=24·8%	..

The figures for the maximum increase of weight in the experiments quoted here vary between 16·8 and 19·9 per cent.; the highest value obtained was 20·6, for an artists' linseed oil. No certain distinction in the increase of weight of linseed oils of different origins could be found, either in these experiments or in another series; the differences are too small to be used to discriminate in doubtful cases. On the other hand, the differences between the results given by the same sample at different times are so large that the method cannot be used for the detection of small adulterations; more gross adulterations are better detected in another manner. The absorption of oxygen cannot also be used for the detection of adulteration, since rosin oil and also rosin absorb considerable quantities of oxygen in the presence of driers.

The chief difference in the behaviour of boiled and raw linseed oil is that the former increases in weight more rapidly than the latter; whether the decrease in weight is also more rapid is yet to be determined. This is probably the case; it would agree with the small durability of a coat of boiled oil as compared with one of raw oil. Further, on the average the values for boiled oil are somewhat lower than those for raw linseed oil.

There is no certain difference between the increases of weight of linseed oil boiled in different manners. In general, with a normal quantity of driers, low numbers indicate a blown or a highly heated oil, which may be adulterated with a non-drying oil, and high numbers indicate an oil prepared in the cold, which may contain rosin or rosin oil; no more exact criticism can be made.

The difference between the behaviour of rosin oil and boiled rosin oil, that is, between the pure oil and the oil containing driers, is exceedingly interesting and worthy of notice; whilst the former loses 15 to 26 per cent. in two months, becoming thick without drying and still continuing to lose in weight after this time, boiled rosin oil behaves similarly to linseed oil (raw and boiled), it increases considerably in weight, dries, and later loses weight. Boiled rosin oil, however, differs from boiled linseed oil in that its increase in weight is greater and that it still absorbs oxygen for 5 to 9 days after it is dry, before it attains its maximum weight. The boiled rosin oils decrease more slowly in weight after drying than boiled linseed oil; this is noteworthy, in drying a portion of the rosin oil must volatilise, so that the actual absorption of oxygen is greater than is indicated by the increase in weight.

Accordingly to Weger, the oils blown at a high temperature, when in a thin layer, begin to increase in weight before the unblown oils; the increase is also at first more rapid and generally comes to an end sooner, that is to say, the oils dry sooner; the increase of weight is, however, always lower than that of the raw oils. The manner of blowing is very important; the temperature and duration of the air current, the size of the air-bubbles, and the proportion of air to oil are of considerable influence on the course of the oxidation. Oils blown in the cold, even for 25 hours, show no apparent alteration in the amount of absorbed oxygen; they dry no more rapidly and are not much thicker than the original oils. It would appear that very little or no oxygen is taken up.

Although a moderately blown oil shows a decrease in the oxygen value as compared with the original oil, yet Weger does not consider the difference large enough to render the process valuable in analytical practice. It cannot be certainly decided whether a thickened linseed oil of unknown origin has been blown or not; thus it cannot be ascertained whether a boiled oil has been made by a process of oxidation or simply by heating. The case is quite different when blowing has been continued until the oil has thickened, the oxygen absorption is then considerably smaller than that of the raw oil.

The question now arises, Can an oil thickened by blowing be distinguished by an estimation of the oxygen absorption from one thickened by boiling? Thick boiled oil shows a similar behaviour to thick blown oil, it does not increase in weight equally with the raw oil. However, the difference between a thick boiled and a thick blown oil appears to be that the former attains its maximum weight later and dries more slowly. The maximum weight of a thick boiled oil is not attained for some time after the oil is dry; the point of dryness is not sharp, and is difficult to recognise.

Weger also examined the oxygen absorptions of hempseed, poppy, and Chinese wood oils. The samples of the two former oils examined gave almost the same increase of weight, which was less than that of raw linseed oil; these oils also have lower iodine values than linseed oil. No definite connection between iodine value and increase of weight could be recognised, otherwise there is nothing calling for remark.

The wood oils differed more widely than linseed oils; from the table an increase of 14 to 16 per cent. may be taken as a mean value; figures considerably under and above this mean value have been obtained. With the exception of experiment 77, the experiments quoted were performed in the summer and old oils were used. But in earlier researches the highest value, 21·7 per cent., was obtained in the winter from a recently imported oil. The great differences in the oxygen values and also in the other constants of the oil, as well as the different manner of their behaviour in gelatinising, etc., appear to have their origin less in differences of the place of production than in the numerous adulterated products which come into the market under the name of wood oil (adulterated in China itself, or also the so-called varnish oil).

The drying point and the time when the maximum weight is attained are often separated by several days in the case of wood oil.

ABSORPTION OF OXYGEN (INCREASE IN WEIGHT) BY HEMPSEED, POPPY, AND CHINESE WOOD OILS.

	Hempseed Oil, Commercial.		Poppy Oil, Commercial.	Wood Oil, Mark P.		Wood Oil, Mark H.			Wood Oil, Turbid.	Wood Oil, Filtered.
Number of experiment .	70	71	72	73	74	75	76	77	78	79
Weight of oil, decimgrms.	308	335	372	483	522	448	233	1175	451	339
Time at which exposed .	Summer 4 p.m.	Summer 6 p.m.	Summer 6 p.m.	Summer 10 a.m.	Summer 12 noon.	Summer 10 a.m.	Summer 6 a.m.	Winter 12 noon.	Summer 11.30 a.m.	Summer 6 p.m.
Increase of weight after										
0½ day .	0= 0·0%	..	..	..	..	..	..	..	..	..
1 ,, .	..	..	..	..	..	..	..	0= 0·0%	..	2= 0·6%
1½ ,, .	..	8= 2·4%	5= 1·3%	2= 0·4%	..	4= 0·9%	8= 2·6%	..	..	..
2 day .	10= 3·2%	30= 9·0%	..	..	..	..	..	23= 2·0%	..	15= 4·4%
2½ ,, .	..	..	12= 3·2%	..	..	..	29=12·4%	..	50=11·1%	36=10·6%
3 ,, .	33=10·7%	43=12·8%	..	4= 0·8%	19= 3·6%	..	37=15·9% (dry)	..	49=10·9%	..
3½ ,, .	..	..	19= 5·1%	..	..	14= 3·1%	..	83= 7·27%	..	..
4 ,, .	42=13·6%	45=13·4%	..	24= 5·0%	32= 6·1%	..	35=15·0%	..	58=12·9% (dry)	..
4½ ,, .	..	..	31= 8·3%	..	..	30= 8·9%	..	..	..	..
5 ,, .	..	42=12·5%	..	44= 9·1%	57=10·9%	45=10·5%	35=15·0%	..	58=12·9%	50=14·8% (dry)
5½ ,, .	..	..	45=11·6%	..	..	..	..	..	..	..
6 ,, .	39=12·7%	41=12·2%	..	59=12·2%	..	58=12·9%	34=14·6%	..	49=10·9%	48=14·2%
6½ ,, .	..	..	50=13·4%	..	..	..	..	195=17·0%	..	..
7 ,, .	37=12·0%	..	..	64=13·2%	72=13·8%	59=13·2%	..	..	..	45=13·3%
8 ,, .	35=11·4%	..	..	65=13·4% (dry)	71=13·6%	63=14·1%	..	..	..	..
9 ,, .	..	..	41=11·0%	..	74=14·1% (dry)	..	28=12·0%	..	..	..
10 ,, .	..	..	..	63=13·0%	73=14·0%	61=13·6%	..	..	..	..
11 ,, .	..	..	..	..	71=13·6%	..	..	..	..	..
12 ,, .	..	..	..	58=12·0%	..	57=12·7%	..	..	..	..
13 ,, .	..	..	..	58=12·0%	..	54=12·1%	..	..	..	..
15 ,, .	..	..	..	..	..	56=12·5%	..	..	..	..
26 ,, .	..	32=9·6%	..	47= 9·7%	..	..	..	..	..	..
36 ,, .	..	..	..	..	60=11·5%	..	..	..	..	..
45 ,, .	..	..	..	44= 9·1%	..	..	..	..	..	..

With regard to the work of Mulder and of Bauer and Hazura, Halphen came to the following conclusions as to the manner of the action of oxygen upon the drying oils:—

1. The acids of the drying oils behave in a similar manner towards the oxygen of the air; differences occur only in the rate at which the oxygen is absorbed, which differences depend upon the relative quantities of linolic and linolenic acid in the different drying oils. The larger the quantity of the former the more rapid is the oxidation.

2. The oxygen absorbed by the oil is not only used to saturate the unsaturated valencies, but at the same time gives rise to the formation of alcoholic compounds.

3. There appears to be complete similarity between the oxidation of the acids of the drying oils and of their salts.

4. When the acids of the drying oils are exposed to oxidation by the atmosphere for a long time, or for some weeks at a temperature of 80° C., anhydrides are formed.

5. The products of oxidation of viscous or resinous nature change into solid substances insoluble in ether, which, when saponified by alkalis, produce compounds soluble in ether. It is thus impossible to decide whether these anhydrides are formed by the condensation of two molecules with elimination of water, or whether they are formed by the elimination of water between the carboxyl group and a peroxide group of the same molecule.

6. It is to be supposed that the acids, which take part in the oxidation of the oil and impart to it its drying properties, are linolic and linolenic acids; oleïc acid, which forms no solid products by oxidation with air, does not take part in the process.

7. The oxidation of the drying oils begins or rather is initiated by the glycerin, but the action appears to depend upon the glycerin united with oleïc, palmitic, and myristic acids. In this manner linolenic acid is oxidised and changes into the anhydride.

For much work upon this subject we are indebted to Livache. After the treatment of the drying and non-drying fatty oils with such substances as would give them the maximum drying properties, they were exposed in thin layers to the action of the air. The exposure lasted two years; each year the increase in weight and the proportion of insoluble fatty acids was determined. From the figures given in

Oil.	Increase in Weight Per Cent.		Insoluble Fatty Acids Per Cent.		Soluble Fatty Acids Per Cent.		
	After One Year.	After Two Years.	After One Year.	After Two Years.	After One Year.	After Two Years.	
Drying Oils—							
Linseed Oil	10·3	7·0	44·3	38·2	40·9	50·2	Somewhat greasy before solidification.
Walnut Oil	9·4	7·6	44·3	45·4	40·9	37·6	Slightly greasy before solidification.
Poppy Oil	8·0	5·3	51·4	44·4	33·0	42·3	Fluid to the touch before solidification.
Cottonseed Oil	6·3	4·5	61·7	57·6	30·9	31·9	Fluid to the touch before solidification.
Beech-nut Oil	6·1	5·0	64·4	55·2	25·6	39·1	Becomes sticky before solidification.
Non-Drying Oils—							
Colza Oil	6·0	5·3	76·7	69·1	25·6	26·1	After two years has become thick, sticky, and transparent.
Sesamé Oil	5·2	4·8	76·8	68·7	22·2	25·0	After two years has become thick, sticky, and transparent.
Arachis Oil	5·7	5·6	72·5	66·6	24·5	29·7	After two years has become very thick, but remains sticky.
Rape Oil	5·8	5·4	77·2	75·8	15·6	19·4	After two years has become thick, sticky, and transparent.
Olive Oil	5·3	5·7	73·1	66·2	15·6	28·9	After two years has become very thick, but remains sticky.

the table the following conclusions are to be drawn. In the first place, oxygen acts upon the drying oils, converting them into solid products, and upon the non-drying oils, turning them slowly rancid, in which process part of the insoluble fatty acids is converted into soluble acids. This transformation increases with the time in proportion to the drying capacity of the particular oil; the higher fatty acids split up after oxidation into lower homologous acids.

In the second place, the examination of the products shows, as also the analyses after two years, that the non-drying oils apparently, in appearance and also in composition, approach certain of the drying oils, as the latter are after the lapse of an equal period of two years. The action is relatively slow, but it should be observed that the same result is attained in far shorter time when the non-drying oils are subjected to a more energetic action of oxygen; these oils are converted under certain conditions into solid products, and experiments have led to favourable results.

Livache gives the following as the conditions which facilitate the drying of a drying oil:—

1. The oil must be refined and clear.
2. It is advisable to allow the oil to stand; a commencement of the oxidation by the absorption of oxygen from the air is advantageous.
3. It is better to employ an oil which has been heated, rather than a raw oil.
4. The drying capacity of a raw or boiled oil is increased by the addition of certain quantities of lead or manganese compounds, or by metallic lead in a finely divided state.

Livache regards the following as the principal factors in causing an increased absorption of oxygen by a drying oil such as linseed oil: (1) the degree of purity of the oil, (2) the time which has elapsed since the extraction of the oil, (3) the manner in which the oil has been stored, (4) the temperature, and (5) the addition of certain substances.

1. *The purity of the oil.*—The oil as it comes from the presses contains water and other impurities, which must be removed from the oil if it is to dry to an elastic mass. This purification is accomplished by allowing the impurities to deposit, by filtration or by chemical treatment. The latter method, which generally consists in the treatment of the oil with sulphuric acid, removes those substances which play no part in the drying of the oil, since they do not unite with oxygen. If recently pressed linseed oil, and linseed oil which has been treated with sulphuric acid and washed with water to neutral reaction, be exposed to the action of the air, the latter absorbs more oxygen than the former.

2. *Age of the oil.*—Linseed oil, preserved with exclusion of air, subsequently absorbs more oxygen the longer it has been kept.

3. *Manner of storage.*—When the oil is stored so that air has access it absorbs a certain quantity of oxygen. Experience has shown that the oil only absorbs oxygen slowly, but that when the absorption has once commenced it proceeds more rapidly. The absorption of oxygen will thus take place more quickly the longer the oil has been exposed to the action of air.

4. *Action of heat.*—The temperature at which the oil is exposed to the action of the air influences the rate of drying. Chevreul showed that linseed oil dried more quickly at 25° to 28° C. than at 15° to 18° C. If at the same temperature in the presence of air, raw linseed oil and the same oil which has been previously heated are spread out in thin layers, the drying is different. Linseed oil, which had been heated at a temperature at which isolated bubbles of gas rose in the oil, dried better, and required only half the time to become hard.

Fahrion believes that when oil is gently boiled in deep vessels, into which the oxygen of the air cannot enter, the transformation of the unsaturated fatty acids is hastened, and products are formed which more quickly absorb oxygen. Chevreul, on the contrary, who heated oil under the same conditions, found that linseed oil which had been heated for 5 hours dried less well than oil heated for 3 hours. There is no doubt that by the action of the continued heating a portion of the glycerin is decomposed, as the smell of acroleïn indicates, and the acids set at liberty; but we know that linoleïc acid dries less quickly than linoleïn. On the same grounds, doubt is cast upon the observation of Mulder that linseed oil which had been heated at 70° to 100° C. dried less well than raw linseed oil. In this connection we may say that (*a*) a moderately high temperature increases the drying of raw linseed oil, and (*b*) raw linseed oil dries less rapidly than the same oil, which has been previously heated, when the two are exposed at the same temperature, but this is only the case when the latter oil has not been heated until it begins to decompose and to form certain quantities of free acids.

5. *Additions of certain substances.*—It has been known for a long time that the addition of certain substances, white lead, litharge, manganese oxide, etc., increased the drying capacity of a drying oil. In certain cases these substances are mixed with the cold oil, in other cases they are heated together with the oil. When lead compounds are used the oil is heated with one or more of them; at the time when free acids are formed by the action of heat, the substances which increase the drying capacity are added, oxidation takes place by means of the oxygen of the lead oxide, and when the process is finished a certain quantity of metallic lead is found in the residue.

This action appears to be important for the following reasons. In the first place, experience shows that lead oleate imparts no real drying property, since it becomes friable; in the second place, the acids of linseed oil dry less quickly than linoxyn. There is, however, no doubt than an oil heated with lead oxide possesses greater drying capacity than a raw oil or an oil without lead oxide. Thus it may be asked whether the drying capacity is not increased by the oxygen of the lead oxide. When it is remembered that a good linseed oil, when exposed to the air, absorbs 16 to 18 per cent. of its weight of oxygen in order to be transformed into a solid dry substance, and that the quantity of lead oxide (red lead or litharge) used in practice does not exceed 3 to 8 per cent. as a maximum, it will be seen that this absorption of oxygen cannot proceed from the lead oxide, since it does not contain more than 0·7 instead of the 18 per cent., which is required; thus the addition of lead oxide can play but a small part in the oxidation of the oil. Also a portion of the lead oxide loses no oxygen, since it forms a lead soap with the oil. However, it is certain that the quantity of metallic lead left at the end of the operation corresponds to some amount of oxidation, and it has been found from a study of the action of oxygen upon the raw oil, that drying proceeds more quickly when the oxidation of the oil has once reached a certain point.

The same observations and conclusions hold good with regard to the drying property imparted by the use of manganese compounds. A just explanation is afforded by the important observation of Chevreul, that linseed oil dries in considerably shorter time when spread out on a clean lead plate freed from every trace of oxide. In this experiment the oil can absorb no other oxygen than that supplied to it from the air, the drying property is thus imparted by the presence of the metal alone. This observation may be further pursued. When a good drying oil is mixed in a closed flask with precipitated lead (obtained by the action of another metal on the solution of a lead salt) the oil acquires good drying properties without being heated; in this case an absorption of oxygen by the oil in the flask is completely excluded. Thus it appears that the small quantities of lead which are dissolved in the oil, *i.e.* by its acids, increase the drying properties.

The drying property due to manganese compounds cannot be directly obtained, but the lead in an oil can be replaced by manganese. It is sufficient to treat an oil, which has been treated with lead, with a manganese salt, *e.g.* manganese sulphate, which by double decomposition produces a lead salt insoluble in the oil, whilst the lead is replaced by manganese. Oil so prepared has very great drying properties, which far surpass those of oil boiled with manganese salts.

The explanation of this increased drying power of a manganese oil is as follows:—When the oil is spread out in a thin layer, so that the air can act upon it, it acquires a darker colour owing to the higher state of oxidation of the manganese oxide induced by the oxygen of the air. In proportion as the oil thickens in drying, this dark coloration disappears; the oxidation of the manganese oxide has been the means of oxidising the oil, and converting it finally into a solid and completely colourless mass.

This coloration, followed by complete discoloration, shows that the manganese oxide has served as the means of the transformation; it has taken up oxygen from the air and transferred that oxygen to the oil. The same process takes place with lead oxide, but less rapidly.

In oils treated with lead or manganese, the lead or manganese plays an intermediate part, absorbing oxygen from the air and giving it up to the oil, thus causing the oil to dry more rapidly than it would without the intermediary. After this explanation the question naturally arises whether other metals can play the same intermediate part as lead and manganese. The simplest method is to heat oil, previously treated with litharge, with metallic salts, the acids of which form lead salts insoluble in oil. Linseed oil boiled with litharge dries in 24 hours when spread out in thin layers, as has been previously stated; if the lead be replaced by manganese, it dries in 6 hours; if by copper, zinc, and cobalt, it dries in 36 hours; and if by nickel, iron, or chromium, in 48 hours. The observation that an oil, in which lead has been replaced by manganese, dries more rapidly than an oil directly treated by a manganese salt is very important. In the industry, we find certain lead and manganese compounds preferred; the choice appears to be influenced by their degree of solubility in the oil under the action of heat; in practice, lead acetate and manganese borate are used by preference, the reason being that these compounds are decomposed in the oil by the action of heat, producing lead and manganese oxides, the favourable action of which is known.

Lead acetate melts in its water of crystallisation at 75° C., loses its water and a little acetic acid at 100° C., decomposes completely into lead carbonate and acetone at 270° C., and also leaves a residue of finely divided lead, which plays a great part in the drying of oils.

Manganese borate is unstable, like all manganese compounds; the affinity of boric acid to manganese oxide is slight, and by the action of heat the acid is set free. Thus the advantage in the use of this compound lies in the easy introduction of manganic oxide into the oil.

We have already seen that unoxidised lead in a very porous condition has very great influence on the drying of oils; it is thus interesting to learn the action of other precipitated metals. Precipitated copper and zinc produce no perceptible increase in the drying properties. Chevreul had already shown that linseed oil dried more rapidly when spread out on unoxidised lead than on copper, zinc, or iron; it appears that the latter metals have no influence on the drying, oil dries on them as slowly as on glass or porcelain.

As regards other substances besides lead and manganese compounds, which are repeatedly proposed, they have no appreciable action. Chevreul had already shown that all these substances not only do not increase the drying capacity, but that certain of them, antimony salts, for example, even decrease it. When a favourable action is occasionally observed with isolated salts it is to be attributed to the action of heat alone.

The drying of every drying oil and boiled oil, apart from differences in the driers used in the latter, depends on many circumstances and conditions. When the same boiled linseed oil is spread on the same clean surface (glass is best), differences in the temperature and moisture of the air and in the light occasion great differences in the time occupied in drying. These conditions have long been known in practice, but more recently they have been scientifically examined. Weger, to whom in conjunction with Lippert we owe the scientific explanation of the processes which occur in the preparation and use of the drying oils, remarks as follows:—The higher the temperature during drying, the more rapidly the oil dries. Pure linseed oil, which dries at the ordinary room temperature in winter in 5 to 7 days, and in summer in 3 to 4 days, requires in a dark drying oven at 50° C. only 12 hours, at 95° C. about one hour, and at 120° C. about 30 minutes. The time of drying of an oil, boiled with manganic hydrate, at the normal room temperature was about 14 hours, at 95° C. 30 to 40 minutes, and at 120° C. 15 to 20 minutes. A litharge boiled oil dried at the normal room temperature in about 9 hours, and at 120° C. in about 20 minutes; a lead-manganese resinate boiled oil dried at the room temperature in about 10 hours, at 95° C. in about 45 minutes, and at 120° C. in 15 to 20 minutes. The industrial practice of drying coatings of certain paints and varnishes in the stove at 50° to 120° C., and recently varnishes at a much higher temperature, not only accelerates the drying but also the hardening of the coats, which is of great importance for many articles.

The influence of light was first studied by Cloëz, who allowed light to pass through colourless, blue, red, green, and yellow glass before it reached the oil; he found that oxidation was most rapid under the colourless glass and least rapid under the yellow. Boiled oil exposed to direct sunlight required for drying only half or one third of the time it required in the shade. This is not only due to the action of light, but, when the heat is not absorbed by alum or Iceland spar, to the combined action of heat and light. The influence of light alone is determined by the fact that boiled oil dries more rapidly in diffused daylight than in a dark box. The same oil dries in very different times at daytime and at night, in summer and in winter. Here again there is the united action of heat and light. Mulder also investigated the influence of sunlight, not in regard to the rapidity of drying in thin layers, but as to the amount of absorbed oxygen and the commencement of its absorption by somewhat thicker layers.

The influence of the amount of moisture in the air is not as considerable as might be imagined. Sheets of glass coated with equally thick layers of the same boiled oil were placed under two bell-jars. In one was a dish of water, in the other strong sulphuric acid. The illumination and temperature were equal. The result was not the same with all boiled oils, not in every case did the oil dry over the sulphuric acid more rapidly than in the moist air. The reverse was frequently the case. Weger could give no general rule; it appeared to be an accident that, of the oils examined, those containing lead or lead and manganese dried more quickly in moist air, whilst manganese oils, on the contrary, dried more slowly than in dry air. It is also true, that when all the conditions are as far as possible the same—the same oil in layers of the same thickness, the temperature, light, and moisture the same—the same drying result is not always obtained. The causes of these differences are not known. Mulder made similar observations; he says: "It is remarkable that when the increase in weight of a drying oil is daily observed, quite different weights are obtained in different conditions of weather, although the temperature and other conditions of the air determinable by ordinary methods do not account for the difference." "The influence of ozone in the experiments was not regarded or considered, but it must be recognised that certain atmospheric conditions, which give rise to greater or smaller appearances and differences of oxidation, are in no way to be accounted for by differences in moisture." Mulder thus supposes certain atmospheric conditions not directly perceptible.

Weger has also investigated the influence of the age of boiled linseed oil upon the time which it requires to dry. In the oil itself, before it is spread out in a layer, there occurs a decrease in drying power, which becomes manifest in certain varieties in the course of time. In general it may be said that lead-manganese oils decrease in drying capacity; lead oils apparently are similar, but observations extending over a long period in this case are wanting; manganese oils generally remain constant, naturally only when changes in atmospheric conditions are not involved, and when the oil is not so loaded with drier that separation takes place in time. The decrease in drying power does not depend on the lead or manganese compounds employed; oils boiled with litharge and pyrolusite, with lead-manganese resinate and with lead linoleate, all show a decrease. With manganese oils the acid of the drier also plays no part. An exact proof of these statements is not easy. In the first place, it would naturally be necessary, if further conclusions are to be drawn and the causes of the decrease in drying power ascertained, at each period to know exactly the quantity of dissolved metal, for as soon as separation of lead or manganese compounds occurs, the drying power must decrease. Thus a decrease in drying power, without separation of driers, has not yet been proved. The results which have led to the conclusions above given cannot readily be made free from objection, since the separate factors which compose the conditions of experiment cannot entirely be regulated. The decrease in the drying power of lead-manganese boiled oils may be rectified by making the oils to dry more quickly at first than is really necessary.

The end-product of the oxidation of linseed oil, whether obtained by the drying of thin or thick layers, by treatment with oxygen or ozonised air, or by the drying of a number of thin layers one upon the other, is the substance called linoxyn by Mulder. It is distinguished by great elasticity, absolute insolubility in water, alcohol, and ether, and by almost instantaneous saponification by potash in the cold. If the fatty acids be separated from the soaps resulting from the saponification of linoxyn, it is found that the solid fatty acids have not been altered, whilst the liquid fatty acids, on the contrary, have changed to greasy substances distinguished by their solubility in water and by the formation of certain salts. Cloëz was the first to investigate this end-product; he found in it fatty acids and an insoluble body. Livache undertook the examination of the solid product, which he subjected to the action of different solvents. The results are as follows:—If the product of the oxidation in the air of thin layers of linseed oil be treated with various solvents, no action can at first be observed. On continued action it is found, with the majority of the solvents, that the transparency is increased, and the mass swells up. If petroleum spirit be used, which has the most energetic action, it is found that a small piece doubles in volume without altering in shape, whilst the liquid is perceptibly coloured. If the oxidised oil be ground with a small quantity of one of the solvents a different action occurs; with petroleum spirit, for example, the transparency increases, the mass swells up and divides into many small particles, forming a kind of salve. If more solvent be now added it is coloured yellow, and the particles, which appear united in a lump, remain in a state of very fine division. With ether, turpentine, acetone, acetic ether, and carbon bisulphide the same appearances are observed: the mass divides more or less quickly, the particles become smaller on protracted grinding, become transparent and swell; on the addition of an excess of solvent the particles separate, and on standing are deposited, whilst the liquid turns yellow. If the grinding with petroleum spirit be continued for a long time, until the solvent is not longer coloured, a separation of the substance into two portions is observed; the one insoluble, swollen, and transparent, the other soluble and imparting the colour to the solvent. When the swollen mass is dried, an elastic substance, which can be rubbed into non-coherent particles between the fingers, is obtained. On evaporation of the solvent a solid residue is obtained, adhering to the fingers and softening at a gentle heat. There is thus a certain similarity between the product of the oxidation of a drying oil and caoutchouc. Both swell in certain liquids, become transparent, and appear to dissolve, but in reality only a portion dissolves. Certain industrial applications are based on this property, as also on the behaviour towards mineral oils. If any non-porous surface be covered with a drying oil, which has been well boiled and tolerably thickened, the entry of the oxygen into the mass of the oil is difficult, it acts only upon the surface and soon forms a solid skin, which protects the oil beneath from the further action of oxygen. This difficulty is avoided by mixing the oil with turpentine, or by applying heat or using very thin layers, of which naturally a larger number is necessary. In the first case, the action of oxygen only takes place after the evaporation of the solvent, and it would be equally good to work with an unthickened oil. In the second case, the labour of obtaining a layer of a definite thickness is increased, and in certain industries it is difficult to use a thin oil, which is absorbed by the object to be covered before it can form a solid and elastic coating.

The industries of the present day use large quantities of solid oxidised oil in preparing impenetrable coatings. Oil is allowed to flow over a woven fabric of several thousand square metres surface, hanging vertically. The very thin layers of oil rapidly absorb oxygen and become solid; the absorption of oxygen is accelerated by conducting the operation in heated rooms. The whole treatment is automatic in action, and at little cost a layer of dried oil several centimetres in thickness is obtained. It can be understood that, with this solid mass and suitable solvents, waterproof fabrics can be made, similar to those obtained from caoutchouc. Either the dried oil, swollen in solvents, is used alone, or mixtures of the finely divided linoxyn ground in an excess of solvent to the required consistency. It is then sufficient to volatilise the solvent, in order to obtain a residue of solid oxidised oil, completely dry throughout, however thick the layer may be, since the oil, when it is used, contains the whole of the oxygen it is capable of absorbing. The " solutions " of oxidised oil may be combined with those of indiarubber, guttapercha, and other substances soluble in the solvents, so that when the solvents have evaporated the residue has the combined properties of all the substances used.

In combination with vegetable fibres, such as cotton, linen, sawdust, and products obtained from these substances, woven fabrics, paper, etc., linseed oil possesses the property of becoming heated; thus danger of fire is often caused when such materials as are used in works for cleaning purposes, saturated with linseed oil, are not removed from the work-rooms or kept in iron vessels. Similar spontaneous combustions occur with other oils, but not so frequently. Kiessling makes the following observations on this subject:—With a sufficiently large surface of contact between a fatty oil, which spontaneously absorbs oxygen, and the air, and suitable protection against external cooling, a considerable amount of heat is developed by the process of oxidation, which, in the case of a fibrous substance saturated with oil, may lead to spontaneous combustion. The quantity of heat produced by spontaneous oxidation depends on the nature of the oil (rape oil shows no, cottonseed oil a small, linseed oil a great, and boiled linseed oil the greatest, rise of temperature), also on the nature of the fabric (silk produces the greatest, hemp the least, rise of temperature, wool and jute are intermediate), on the external temperature (the higher this is the greater is the development of heat), and on the light. The activity of micro-organisms is without effect in this case; the process is purely chemical.

Mackey is not of the same opinion in regard to the action of oil upon cotton-wool. He remarks that the fire insurance offices fix the premiums of the cotton spinners according to the nature of the oil used in preparing the cotton; the oil must not contain more than a certain percentage of unsaponifiable matter, and its point of ignition must not lie below 171° C. It is, however, better to determine directly by experiment the danger of spontaneous ignition. For this purpose cotton-wool saturated with the oil is heated in a bath at 100° C., the bulb of the thermometer being in the middle of the cotton-wool. By taking the temperature after heating for a certain period it is found whether the oil inclines to spontaneous ignition. Experiments with cottonseed and olive oils showed that the temperature rises far less with neutral oils than in the presence of free fatty acids. For example, the temperature of the cotton-wool after 3 hours was, in the case of olive oil, 100° C., but with free fatty acids, 192° C. After some time the temperature again sinks. Frequently, in the presence of much free fatty acid, the cotton-wool was charred; but ignition was never observed. The cause of the rise of temperature is not to be found exclusively in the oxidation of the fats and fatty acids, since the temperature of slag-wool heated with oil or fatty acids did not rise above 100° C. Thus the cotton must be attacked by the fatty acids, and the temperature thereby increased.

CHAPTER II

THE PROPERTIES OF AND METHODS FOR OBTAINING THE DRYING OILS

THE number of the drying oils, which are extensively used in the industries, is small as compared with the number of non-drying oils, and even amongst these—candle-nut oil, cedar-nut oil, hempseed oil, wood oil, cherry kernel oil, linseed oil, poppy oil, walnut oil, castor oil, sunflower oil, grapeseed oil—linseed oil is the only one which, apart from the locally used hempseed oil, is important in the preparation of varnishes and paints. Candle-nut, cedar-nut, walnut, sunflower, and grapeseed oils are so rarely produced, and in such small quantities; hempseed oil is used in Russia alone; wood oil is much too dear, and the imports insufficient; castor oil is excluded from extensive use for these purposes because its drying properties are insufficient, it is, however, highly valued for the preparation of Turkey red oil, of soaps, lubricants, and recently of an indiarubber substitute. There can be no doubt that candle-nut oil, and especially wood oil, would become of great importance if they could be produced in quantity and at a price not far removed from that of linseed oil. Under present conditions linseed oil is all important, thanks to its good properties and low price.

CANDLE-NUT OIL.

Bankulöl, Lichtnussöl, Lackbaumöl.
Huile de noix Bancoul, huile de Bancoul.

The source of this oil is the candle-nut from *Aleurites triloba* and *A. moluccana,* which could be placed on the market in very large quantities from Guadeloupe, Martinique, New Caledonia, Tahiti, and Réunion, but which is not yet to be obtained regularly. Not only the cheapness of the raw material, but the good qualities of its oil, recommend its introduction to oil works.

According to Wiesner, the seeds contain 50 to 60 per cent. of oil. The French Marine Ministry states that 100 kilos. of nuts produce, on an average, 33 kilos. of kernels, 100 kilos. of which give 66 kilos. of oil. Thus 450 kilos. of nuts would be required to produce 100 litres (91 kilos.) of oil. In Tahiti the nuts cost 150 francs per ton of 1000 kilos., freight amounts to 80 francs per ton. The kernels are sold at 400 francs per ton; thus, since 100 kilos. of kernels produce 66 kilos. of oil, 100 kilos. of the latter would cost 60 francs, without reckoning the cost of pressing. The price offers no advantage in consideration of the low cost of linseed oil.

Properties of candle-nut oil.—The cold-pressed oil is pale yellow, almost white; the hot-pressed oil is brown. The latter has a nauseous, the former an agreeable, odour. It dries in the same time as linseed oil, and by similar treatment may be so altered in drying properties that it may be used as boiled oil of fairly considerable drying power. It burns well; excellent results have been obtained in this respect by various establishments in Paris.

To the writer's knowledge, analytical examinations of this oil have not yet been made; the writer's own researches have been restricted to the applicability of the oil for varnishes and paints. Small quantities of the brown oil were boiled with litharge, red lead, lead acetate, and manganese borate. The oils boiled with lead compounds were all very dark in colour; at 140° C. the oil effervesced; it was maintained at this temperature for a short time only, which was necessary owing to the small quantity of oil treated. The smell of the boiled oil was quite similar to that of the raw oil, but slightly more unpleasant. The oil boiled with manganese borate was almost unaltered in colour. In the drying tests, as compared with well boiled linseed oil, there was a considerable difference: the boiled candle-nut oils dried at least 4 hours before the boiled linseed oil, the raw oil also dried more quickly than raw linseed oil. At 325° C. it evolved vapours of very strong, unpleasant smell, which did not take fire. After a loss of 20 per cent. of volatilised fatty acids there resulted a thick, soft mass, as with linseed oil, but the colour was almost black, which was due to the presence of a large quantity of mechanically admixed impurity. The same experiments were performed with the white oil. Almost all the oils boiled with lead compounds were strongly coloured, whilst the oil boiled with manganese borate was only somewhat yellow. In the drying tests the same advantages over linseed oil were found, the candle-nut oils dried several hours earlier. On the other hand, when the pale candle-nut oil was heated to 325° C. the peculiar result was observed that, after a loss of about 20 per cent. of volatile fatty acids, the oil became completely colourless, forming a thick water-white syrup.

In regard to the durability of paints made from coloured earths or metallic oxides and this oil, the same results were obtained as with boiled linseed oil. Since, however, candle-nut oil is always dearer than linseed oil, and rarely comes into the market, there appears to be no prospect of its general employment.

Technical uses.—Like linseed oil, for varnishes and paints; as a burning oil.

De Negri has recently examined candle-nut oil from the seeds of *A. moluccana*; he obtained by extraction 62 per cent. of oil. It dried well, but not as well as Chinese wood oil from the allied plant *A. cordata.* The following constants were obtained:—

	Oil extracted with	
	Ether.	Petroleum Spirit.
Specific gravity at 15° C.	0·926	0·921
Solidifying point	– 18° C.	still fluid
Melting point of fatty acids	20°–21° C.	...
Solidifying ,, ,,	13° C.	...
Saponification value	187·4	184·0
Iodine value	139·3	136·3
Iodine value of fatty acids	144·1	142·7
Refractometer (Zeiss)	...	75·5–76

The colour reactions obtained by De Negri were little characteristic. The constants found for wood oil are not different from those of candle-nut oil and from those of a candle-nut oil examined by Lach, which most probably was a mixture of oils from several varieties of *Aleurites.* When exposed to sunlight for 25 days in a sealed glass tube the oils remained fluid, whilst expressed wood oil solidified under the same conditions. De Negri observes that wood oil extracted by solvents does not solidify under the action of sunlight, but only the expressed oil. The polymerised substance from the latter oil melts at 32° C.; the iodine value has fallen from 159 to 154; when heated for 100 hours in the dark at 100° C. it does not change, but on cooling remains liquid. Candle-nut oil extracted by carbon bisulphide always remains fluid, whilst wood oil extracted by the same solvent solidifies.

CEDAR-NUT OIL.

Cedernussöl.

According to Kryloff, a good drying oil is obtained in Siberia from the fruits of the cedar, or rather from the kernels of the seeds, and is already made on the large scale. The following are the constants of the oil:—

Specific gravity at 0° C.	0·9320
Solidifying point	20° C. (?)
Iodine value	149·5–150·5
Reichert-Meissl value	2·0
Hehner value	93·33
Saponification value	191·8
Acid value	1·09

From the mixture of insoluble fatty acids pure palmitic acid could be separated by crystallisation. From the filtrate from this acid there were obtained by oxidation an acid of the same formula as tetrahydroxystearic acid (melting point, 165° C.) and a trihydroxystearic acid (melting point, 140° C.). Thus this oil differs little from linseed, hempseed, and similar oils; it belongs to the class of the good drying oils.

HEMPSEED OIL.

Hanföl.
Huile de chanvre, huile de chènevis.

The fruit of the hemp plant, *Cannabis sativa*, from which the oil is obtained, is a nut; it comes into commerce freed from the capsule, which surrounds it like a sheath and is fissured at the anterior end. The fruit is oval, 3 to 4 mm. long, 2 mm. wide, somewhat compressed on the dorsal side, unilocular, bivalved, does not split open, and contains one seed. The capsule is thin and hard, externally greenish or greyish brown, marked like a net, smooth; it consists of two layers readily separable from one another, of which the outer is pale green and the inner brownish green. The seed has the same form as the fruit, which it completely fills, is adherent, and covered by a thin green skin. The embryo is white, it consists of parenchymatous cells, which contain oil and aleurone granules; it is bent into the shape of a hook, the radicle being bent round towards the inside of the thick cotyledons, with which it is of equal length. When crushed the seeds emit a peculiar odour; they have a mild oily taste, and contain 30 to 35 per cent. of oil. The yield of oil by expression is about 28 per cent., by extraction 30 to 32 per cent. The seeds have the following composition:—

	German Hempseed, per cent.	Russian Hempseed, per cent.
Organic matter	54·30	54·95
Ash	3·45	4·50
Water	8·65	9·13
Oil	33·60	31·42
The organic matter contains proteïns	15·95	15·00

Hemp seed for sowing must be quite ripe, not more than one year old, and 90 per cent. should be fertile. The best seed is obtained by threshing; ripples do not yield a product of equal value. Seed for sowing is frequently obtained from Russia and its Baltic provinces, where it is better to allow it to ripen, since the female plants yield a less fine fibre. The Chinese "giant-hemp" is a larger and coarser fibred variety, as is also Indian hemp, from which hachisch is obtained.

Properties of hempseed oil.—The recently expressed oil is greenish yellow, when extracted by petroleum spirit or carbon bisulphide it is brownish yellow, freshly extracted by ether it is deep green. The green colour of the oil recently expressed or extracted by ether is lost after some time, when the oil becomes brownish yellow. The odour and taste are the same as of the seed, *i.e.* mild.

Specific gravity at 15° C. .	0·9255	(Souchère).
„ „ „ .	0·925–0·931	(Allen).
„ „ „ .	0·9276	(Fontenelle).
„ „ „ .	0·9270	(Chateau).
Behaviour on cooling:—	Thick at − 15° C., solid at − 27·5° C.	
Melting point of the fatty acids	19° C.	
Solidifying „ „	15° C.	(Hübl).
Saponification value . .	193·1	(Valenta).
Iodine value . . .	143	(Hübl).
„ „ of the fatty acids	122–125·2	(Morawski and Demski).
Viscosity at 15° C. . .	9·6	(water = 1).
„ „ 7°·5 C. . .	11·6.	

Hempseed oil dissolves in 30 parts of cold alcohol and in every proportion in boiling alcohol. In the cold it separates a "stearine," which dissolves in 12 parts of hot alcohol. One volume of the oil requires two volumes of ether for solution.

Composition.—The elementary composition of the oil is

Carbon	76·05	per cent.
Hydrogen	11·35	„
Oxygen	12·60	„
	100·00	

The liquid fatty acids consist principally of linolic acid, with small quantities of linolenic, isolinolenic, and oleïc acids.

The oil dries less rapidly than linseed oil.

Colour reactions.—When boiled with caustic soda solution (specific gravity, 1·340) it gives a brownish-yellow hard soap; linseed oil gives a yellow liquid soap. Sulphuric acid colours hempseed oil, as also linseed oil, an intense green. A mixture of equal parts of water, strong sulphuric acid, and fuming nitric acid gives, when mixed with five times the quantity of oil, a green coloration, at once changing to black, and after 24 hours to a reddish brown. Strong hydrochloric acid colours fresh oil grass-green, older oil yellowish green.

Applications of Hempseed oil.—

Alimentary: the freshly expressed oil is used like olive oil and other oils and fats.

Technical: as a burning oil, in soap making, for varnishes and paints.

WOOD OIL.

Chinese Wood Oil, Tung Oil.
Chinesisches Holzöl, Oelfirnissbaumöl, Tungöl.
Huile de bois.

Chinese (or Japanese) wood oil is obtained from the seeds of *Elæococca* or *Aleurites cordata,* which is a native of China and Japan. It is extracted by hot and cold pressing.

The older statements as to the nature of this oil, which is endowed with very great drying capacity, are as follows:—The cold pressed oil is pale yellow, the warm pressed oil dark brown; it is a thick oil of high specific gravity, it does not solidify in the cold. The oil, which possesses still greater drying power than candle-nut oil (from *Aleurites triloba*), is used in China and Japan as a natural varnish, especially for varnishing and preserving wood-work, Chinese ships, etc. The oil is not to be confounded with the balsam wood oil, gurgun balsam, obtained from *Dipterocarpus turbinatus*, which possesses quite other properties; gurgun balsam is, however, adulterated with wood oil. The examination of the oil by Cloëz shows that it consists of two glycerides, ordinary oleïn, and the glyceride of elæomargaric acid, from the latter of which, after saponification, elæomargaric or margarolic acid, $C_{16}H_{30}O_3$, is obtained. This statement was inherently improbable because of the constitution of the acid, as also, considering the great drying properties of the oil, the statement that the oil changes in the air to a fat melting not below 32° C. In regard to rapidity of drying, it surpasses linseed oil; the hot pressed oil does not appear to dry so rapidly as the cold pressed. It is rapidly saponified on heating with alcoholic potash; and having regard to the amount of potash required for saponification (21·1 per cent.), it would appear to contain a hitherto unknown fatty acid. The yearly export from Hankow, a town on the Yangtse-Kiang in the interior of China, is about 200,000 piculs (1 picul = 122·3 lbs. = 60·479 kilos.) to other parts of China.

Moritz Kitt investigated Chinese wood oil. The samples were obtained (1) direct from China, (2) from the firm of Carlowitz in Hamburg. Sample (1), marked "Yutshing," was pale yellow; the following constants were observed:—

Specific gravity at 15° C. . .	0·9413
Saponification value . . .	190·7, 191·0
Iodine value	157·5, 158·4

Acid value	3·9
Melting point of fatty acids . .	35–39°·5 C.
Saponification value of fatty acids .	197·3, 197·8
Yield of insoluble fatty acids . .	82 per cent.

The fatty acids formed a yellowish-brown crystalline mass. By the action of bromine upon the fatty acids, dissolved in glacial acetic acid and cooled by ice, liquid bromine addition compounds were principally formed; in small quantity a solid bromide was obtained, which, after repeated recrystallisation from alcohol, formed fine needles melting at 129° to 130° C. In the oxidation of the fatty acids in alkaline solution by potassium permanganate they remained partly unaltered, trihydroxystearic acid was formed in small quantities, and also acids soluble in water similar to linusic acid; lower fatty acids, acetic and carbonic acids were also produced.

The oil (2) was somewhat darker in colour and more viscous. The following values were obtained:—

Specific gravity at 15° C. . .	0·9439
Saponification value . . .	190·9, 191·4
Iodine value	154·6
Acid value	6·95
Melting point of the fatty acids .	35°·5–40° C.
Saponification value „ „ .	196·4
Iodine value „ „ .	169·5
Yield of insoluble „ „ .	85 per cent.

A portion of the oil (2), which had solidified by exposure to light, had the

Saponification value . . .	191·0, 191·8
Iodine value	155·2, 156·1

practically the same as the original oil. A larger quantity of the oil (2) was heated in a porcelain dish and stirred with a thermometer. At 282° C. it solidified. The gelatinous mass was only partially soluble in benzene; it was completely saponifiable by alcoholic potash. The fatty acids separated from the soap solution were dark brownish yellow with a greenish fluorescence, and were viscous. After several days they solidified to a crystalline mass melting at 41° to 46° C. They had the following properties:—

Saponification value (Alkali Blue as indicator)	198·8
Acid value	191·0
Iodine value	107·6, 108·4
Acetyl acid value	186·9, 187·6
Acetyl saponification value	204·1, 206·4
Acetyl value	18·0

The change into the solid state is due rather to polymerisation than to a sudden absorption of oxygen. The low ether value of the fatty acids (7·8) indicates the presence of anhydrides of lactonic character. On the other hand, the acetyl value (18) indicates only a small quantity of hydroxy-fatty acids. If the oil be heated on a sand bath for about 2 hours at 150° to 180° C., whilst a current of air is led in, a viscous oxidised oil is obtained, which contains hydroxy-fatty acids in larger quantity. The fatty acids separated from such oxidised oil melted at 39° to 45° C., and had the following properties:—

Saponification value	199·5
Acetyl acid value	173·5
Acetyl saponification value	214·6
Acetyl value	41·1

According to De Negri and Sburlati, the seeds from which wood oil is obtained contain 53·25 per cent. of oil, of which about 42 per cent. can be obtained by pressing. The oil is clear, somewhat yellow in colour, and of peculiar smell, resembling that of castor oil. It is without taste, and dries in 24 hours in the air. It is soluble in ether, petroleum spirit, and chloroform, and insoluble in cold absolute alcohol. By solution in carbon bisulphide and evaporation of the solvent the oil is changed into a crystalline mass.

According to older statements, wood oil has the following constants:—

Specific gravity at 15° C.	0·940 (Davies and Holmes).
,, ,, ,,	0·936–0·941 (De Negri and Sburlati).
Melting point of the fatty acids	43°·8 C. (De Negri and Sburlati).
Solidifying point of the fatty acids . . .	31°·2 C. ,, ,,
Saponification value .	155°·6–170° ,, ,,
,, ,, .	211 (Davies and Holmes).
Iodine value . . .	159–161 (De Negri and Sburlati).

The amount of free acid found in a sample examined by De Negri and Sburlati was 1·18 per cent., calculated as oleïc acid.

Very recently Jenkins has made the following communication concerning Japanese wood oil:—

Specific gravity at 12° C.	0·9385
Point of solidification	below 17° C.
Iodine value	165·7
Saponification value	194
Hehner value.	96·4
Unsaponifiable matter	0·54 per cent.
Temperature rise in Maumené's test .	372
Melting point of the fatty acids . .	37° C.
Solidifying point ,, ,, . .	34° C.
Iodine value of ,, ,, . .	150·1
Viscosity in Redwood's apparatus at 12·5° C.	41

If 5 grms. of oil are mixed with 2 c.c. of carbon bisulphide and 2 c.c. of sulphur chloride and the mixture stirred in the cold, a thick lumpy jelly is formed. At the boiling point of water a skin begins to form on the surface of the oil in 15 minutes, in 2 hours it completely covers the oil; after 4 hours' heating the increase in weight is 1·44 per cent. In the elaïdin test a dark brownish-red mass is formed, consisting of a heavy, almost solid, portion and an oily residue;

when stirred up the whole is very viscous. With Becchi's reagent a deep reddish-brown mass is produced; strong sulphuric acid converts the oil into a solid black mass; nitric acid of 1·4 specific gravity forms with the oil a soft mass which, on standing, becomes darker and harder. When 5 c.c. of a saturated solution of iodine in chloroform are added, whilst stirring, to a solution of 1 grm. of oil in 5 c.c. of chloroform, the mixture solidifies in 2 minutes to a stiff jelly; when 2 grms. of oil are used, the mass is so hard that it can be powdered. When heated in the absence of air to 180° C., small quantities of vapour are given off, but the oil does not become more viscous; above 250° C., the oil changes to a solid clear elastic mass, which is insoluble in the ordinary oil solvents, and does not again melt at the temperature of its formation.

In regard to the drying of the oil, the following observations have been made:—If a very thin layer is spread upon glass it dries in a closed space in about 24 hours, with a whitish appearance, so that the combination of oil and glass is no longer transparent but resembles ground glass. In thicker layers it requires several days to dry off. If the coating is exposed to the light, even thick layers dry in a few hours; the oil which has been heated alone, without additions, shows on drying the same whitish appearance, whilst oil which has been heated with litharge dries clear and bright, as boiled linseed oil does under any conditions. The oil shows the greatest peculiarity after drying; whilst raw and boiled linseed oil and the other drying oils form a slight skin which gradually thickens from below, wood oil dries uniformly throughout, so that the lower layers are as dry as the upper. Even quite thick layers may be removed as a soft but still somewhat brittle coating, which is uniform throughout. The slight adhesion of the dried coating to the smooth surface of the glass is remarkable.

Tucker also examined wood oil. He found

Specific gravity	0·937
Saponification value	197
Iodine value	163
Melting point of the fatty acids . .	43·84° C.
Solidifying ,, ,, . .	31·4° C.

The oil mixes with chloroform, ether, petroleum spirit, and fatty oils in every proportion, but not with alcohol. The formation of a jelly, already observed by others, on mixing 5 grms. of wood oil with 2 c.c. of carbon bisulphide and 2 c.c. of sulphur chloride, does not occur when the oil has been mixed with the oil of *Dipterocarpus turbinatus*, a common adulteration.

Ferdinand Jean remarks concerning wood oil: This oil is obtained by pressing the seeds of *Elæococca vernicia*, a tree belonging to the family of the Euphorbiaceæ, which grows in China and Cochin China. The oil should not be confounded with the wood oils of Cochin China obtained by puncturing the trees of the family of the Dipterocarpeæ. The constants of the oil are as follows:—Specific gravity at 15° C., 0·940 (the density of the three oils generally designated wood oil, white, light, and black, varies between 0·960 and 0·966); refractometer, 1°·80; point of ignition, 260° C.; acidity calculated as sulphuric acid, 0·784 per cent. Sulphuric acid produces a black coloration. The elæococca oil is golden yellow and clear; at 18° C. it becomes considerably thicker, without losing its transparency. When heated for some time at 200° C. it becomes suddenly solid, and forms a transparent jelly which does not adhere to the finger; this property depends on the absorption of a very large quantity of oxygen. It has extraordinary drying powers. Under the influence of light it solidifies without air being necessary. Upon a smooth metallic surface it produces a transparent varnish in a short time. It is completely saponifiable, and contains 72 per cent. of fatty acids, which melt at 44° C.

The writer's own experiences with Chinese wood oil now follow. Wood oil has recently become an article of commerce. Two varieties, Canton and Hankow oil, are offered by the Hamburg importers, according to the arrivals. Although the price is still high, on account of its peculiar and signal property of drying throughout in a short time under the influence of air and light, it has already obtained an entrance into the manufacture of varnishes and paints. There is no doubt that when thorough research has been made a number of applications will be found for it. Thus Chinese wood oil is used for oiling floors on account of its hardness, also in the manufacture of waterproof materials it has been made into a substance similar to oilcloth and distinguished by great elasticity.

The samples of wood oil used in the author's experiments were partly Hankow and partly Canton oil. The two varieties were tolerably similar in consistence and colour; they had the same peculiar odour, recalling dripping, by others thought to smell like bugs. Some samples were turbid, others quite clear and transparent. In most cases the turbidity disappeared on heating, and both varieties of oil—Hankow and Canton, turbid or bright—dried with a whitish appearance not shown by the heated oil. The formation of this translucent whitish layer on drying is probably due to the presence of mucilage and albumin. Thus the raw oil cannot well be employed if it is to be used in the same manner as linseed oil; it must be heated with several per cent. of lead oxide (litharge or red lead), for without this addition it gives a non-transparent layer. The same inconvenience would not be caused if the oil were used in paints, but it is necessary to heat it in order to obtain increased drying power. When the oil is heated alone, or with lead or manganese compounds, it is necessary to keep the temperature carefully below 200° C., otherwise—especially with manganese compounds (manganous borate)—thickening occurs, and in a very short time complete gelatinisation, which makes the product incapable of further use. The oil should thus only be heated to 160° or 180° C. as a maximum. The pan should then be withdrawn from the fire, or the fire extinguished and the driers then added; this treatment is sufficient to give to the oil the desired drying power and to remove the above-mentioned drawback. Pigments ground with oil treated in this manner produce paints of excellent properties, which in a short time dry hard throughout, and not as the paints made with linseed oil—dry only on the surface, but in the lower portions of the layer still soft after a long time. Paints which dry hard are in demand, and the higher price is unimportant in their case.

This same important property makes wool oil also suitable for the manufacture of oil varnishes; it cannot, however, replace the oils used in spirit varnishes, because it is absolutely insoluble in alcohol. In using this oil for oil varnishes, it is of the first importance to consider that at a temperature of about 200° C., when heated with lead or manganese oxides, it thickens considerably and even gelatinises. Thus that method of manufacture must be used in which the completely melted and cooled copal is dissolved in the hot oil at a temperature below 160° C. Then the varnish can be boiled with dryers without fear of gelatinisation. The combination of wood oil with linseed oil gives very good results, and especially in the case of varnishes for outside use; the former oil imparts hardness, the latter elasticity. The smell of Chinese wood oil is very peculiar; it is retained by dried coatings so persistently that even material similar to oilcloth, which has been dried in hot air, retains the odour even for months, just as oilcloth always smells of linseed oil even after the lapse of years. Naturally, this peculiar dripping-like smell is perceptible with the varnishes made from this oil, and it appears necessary to remove it entirely or to reduce it very considerably. It is hardly sufficient to hide the smell by an essential oil, because when this has evaporated the smell again appears. Other means must be found; such are—shaking with a dilute solution of potassium permanganate, with a filtered solution of bleaching powder, filtering over bone black, mixing with potato starch and filtering after long standing. It is said to be possible, by the process of Bang and Ruffin, to obtain tolerable freedom from smell by the action of dry air. The oil is warmed in a suitable pan to 50° C., which temperature must not be exceeded; a current of air is then introduced by means of a fan or blowing machine, and continued at least 6 to 8 hours, after this period the oil is considerably improved in odour and can be used without hesitation for varnishes.

It should not be omitted to state that wood oil should not be regarded as a substitute for linseed oil, but as a special drying oil from which can be made varnishes, oils, and paints with the special property of drying rapidly and completely throughout. On this account it ought not to be necessary to lay great stress on making the oil free from smell; the characteristic smell should rather serve as a peculiarity of the substance, especially as it is not unpleasant.

The Consular Reports of the United States contain the following information concerning Chinese wood oil. "The Chinese oil tree, *Aleurites cordata*, belongs to a family very common in China, to which the name 'tung' is given. The oil tree itself is called 'ying-tzu-tung,' so called after the shape of its fruits, 'ying' meaning bottle. It grows to a height of 10 to 15 feet in stony ground, and chiefly in Hunan, Hufeh, and Szeihuen. The fruits, which are gathered in August and September, contain five to seven large and poisonous seeds, from which the oil is expressed.

Hankow is the principal place of export, from which, in 1895, 290,631, and in the first 9 months of 1896, 188,556 piculs of oil were exported. The oil is obtained in a similar manner to earth-nut oil. The nuts are collected, dried and placed in an ordinary Chinese reis-kwo (a shallow iron vessel 2 feet in diameter), which is put over an open fire. They are then ground to powder by stones and pressed in wooden presses, when the oil flows out; generally it becomes bright only after some time. It is principally used for varnishing boats—every Chinese boat receives one or two coats—in the preparation of varnishes and waterproof materials, for medical purposes, and in western China as a burning oil. Mixed with lime, it forms a good cement for earthenware. The mass from which the oil has been extracted is burnt; it produces a valuable soot, from which Indian ink is made. At the place of production the oil costs about 2·5 taels (dollars) per picul, but in Hankow already double as much. In the fresh state it is said to be extremely poisonous. It appears to be largely adulterated in China with cheaper oils, especially cottonseed oil."

A careful examination of all the researches here quoted shows that the statements regarding the properties of Chinese wood oil agree only in that the oil dries well and that it gelatinises at a certain temperature. The cause of the difference in the analyses and estimation of the constants lies, in the first place, in the great difference in the oils themselves, which are not produced from the same material worked in the same way, and in the manner of the investigation. The time is within sight when the properties of wood oil will be definitely known.

Rosenblum and Rideal obtained from wood oil substances which they call "tungates." Wood oil is saponified by potash, soda, or ammonia; by double decomposition of the alkaline soaps with solutions of metallic salts, metallic soaps are obtained, which can be used as driers. These metallic soaps are readily soluble in linseed oil; much smaller quantities produce the same effect as resinates and linoleates. If ozone be blown through oil containing 0·7 per cent. of "tungates" at 130° F., a viscous and colourless oil, which dries very well, is obtained. The metallic salts are also soluble in turpentine.

Substance resembling indiarubber from Chinese wood oil.—This substance, which possesses considerable hardness, good face, resistance to changes of temperature, the action of chemicals and weak acids, and which is said to be used in the manufacture of varnishes, consists principally of wood oil, manganese borate, and Caucasian pyrolusite, which contains a large percentage of manganese and a small percentage of oxygen. The oil is mixed, at about 150° C., with 0·5 to 2 per cent. of manganese borate and 1 to 3 per cent. of Caucasian pyrolusite, both in powder; it is then heated to 200° C., with continual stirring, and maintained at this temperature for 1 to 1½ hours, until complete oxidation occurs, *i.e.* until the mass has solidified to a rubber-like substance. The proportion of pyrolusite and manganese borate is chosen according to the drying capacity required, which can thus be easily regulated.

PUMPKINSEED OIL.

Kürbiskernöl.
Huile de pepins de citronelle.

The material from which the oil is obtained is the seed of *Cucurbita pepo*, L. The oil is much used in Austria-Hungary as an edible oil; among the edible oils it stands next to olive oil in price. Since the constants of this oil are not yet widely known a number of pure oils were examined, some of which were expressed from the seeds and some obtained from commercial sources. The oils obtained by cold and hot pressure gave almost the same values. The following are the extreme figures:—

Specific gravity	0·923–0·925
Iodine value (Hübl)	122·8–130·7
Saponification value	188·4–190·2
Refractometer at 35° C.	70·0– 72·5
Melting point of the fatty acids—	
Commencement	26·5– 28·5
End	28·4– 29·8

The following oils might be used as adulterants—linseed, sesamé, cottonseed, and rape. In a commercial oil which possessed the under-mentioned constants Voda, to whom this examination is due, ascertained the presence of about 36 per cent. of cottonseed oil and 64 per cent. of pumpkinseed oil.

Adulterated Sample.

Specific gravity at 15° C.	0·92527
Iodine value	119·6
Saponification value	190·2
Melting point of the fatty acids	30°·5 C.
Refractometer at 35° C.	69·2

Becchi's reagent gave a positive result.

Linseed Oil.

Leinöl, Leinsamenöl.
Huile de lin.

The raw material from which linseed oil is obtained is linseed, the seed of *Linum usitatissimum*, the flax plant, which is cultivated principally on account of its fibre, but in some countries only for its oleaginous seeds.

The chief flax-growing countries, the Baltic provinces of Russia, India, Egypt, North America, and recently also South America (Brazil), also produce the largest quantities of commercial linseed. Commercial linseed is intended either for sowing or for the extraction of oil. The species of seed used for sowing is heavy, ripe, fresh, and still capable of germination; for pressing, the smaller seeds are used, which may be brought from the fields whilst still unripe, or have lost their power of growth in consequence of long or careless storage. Unripe linseed is chiefly used for crushing, it is obtained as a by-product of the flax manufacture. The flax only produces a satisfactory fibre when it is harvested before the seeds are ripe. The seeds which are then obtained are quite suitable for crushing, but not for sowing. For technical purposes, seed for crushing alone comes into consideration, since fresh seed for sowing is only produced in small quantity, and in those regions where linseed oil is used as a food or in the preparation of foods.

Each fruit of the flax plant contains ten seeds. The seeds attain a length of 3·5 to 5 mm., are much flattened, oval in outline, with a smooth surface and greenish-brown to brown colour, of a slight but unpleasant smell. At the small end is the hilum. Under the microscope the surface does not appear smooth, but covered with delicate indentations. The average weight of a single seed is 0·3 to 0·5 mgrms. The seeds suitable for sowing are about 5 mm. long, and over 0·4 mgrms. in weight. Good linseed, which has become incapable of germination, is of the same size and weight, and such seed is to be preferred for crushing before unripe seed, which is smaller, lighter in weight, and generally very greenish in colour. The anatomical examination under the microscope affords a means of distinguishing ripe from unripe seeds. In the seeds three parts can be clearly distinguished—the husk, the embryo, and albumin. The albumin adheres closely to the husk. In the interior of the seed lies the greenish-yellow embryo, with the radicle about 1 mm. long surrounded by the whitish albumin. The hard and brittle husk is made up of five layers. The external layer is a skin composed of colourless cells, the outer layers of which swell up considerably in water. Next follows a layer of cells consisting of soft elements, in which is enclosed a layer of tissue formed from long sclerenchymatous cells, which latter give the husk its hardness and density. The next following fourth layer of tissue has great similarity with that lying beneath the epidermis, it also is composed of soft cells joined together. The fifth and innermost layer consists of grouped polygonal cells flattened parallel to the surface of the husk, which contain a brown granular substance. This layer gives the husk of the linseed its peculiar brown colour. In grinding, the tissues of the husk, down to the sclerenchyma cells and the elements of the innermost skin, are destroyed. Portions of the third and fifth layers are generally to be found whole in linseed meal; they can always be used to recognise this meal as such or when pressed, and also when the meal is used as an adulterant of other meals. The albumin consists of soft polyhedral cells, which contain drops of fat and aleurone grains when the seed is ripe; in the unripe state they also contain starch in small granules. The diameter of the cells is 0·009 to 0·013 mm.

The tissues of the embryo are made up principally of cells, which in form, size, and contents agree with those of the albumin. Among these cells lie the elongated and compressed rudimentary organs. The fresh seeds, when some time immersed in water, become coated with a transparent jelly, which dissolves in the water when the immersion is of long duration.

The seeds contain about 33 per cent. of oil, of which approximately 26 per cent. is technically extracted. According to Chevallier and Lassaigne, linseed contains the following amounts of oil, according to the country of growth :—

Flanders	39 per cent
Russia	35 „
Nantes (Picardy)	33 „
Brittany, Strassburg, La Rochelle, Rochefort	31 „
Hamburg	30 „
St. Petersburg	29 „

The seeds also contain about 8 per cent. of water, 25 per cent. of albumins, and 4 to 5 per cent. of mineral substances.

In regard to linseed for sowing, the seed from northern countries has the reputation of producing good fibre, that from southern countries a better fruit. Thus the Baltic province of Russia and East Prussia are renowned as producers of seed for crushing; Riga, Windau, Pernau, Libau, Memel, and Königsberg supply not only Germany, Belgium, and Southern Europe, but also North and South America and Australia; the Riga seed is accounted the best. The Tyrol, Saxony, Bavaria, Holstein, Mecklenburg, and Hungary, however, produce equally good seed; Egypt, North America, and India produce large quantities. The important characters of linseed are its source, fertility, and freedom from the seeds of weeds. The origin of linseed cannot well be ascertained, except from the markings on the seamless bags or on the barrels in which Russian seed (for sowing) is generally packed. Seed for oil mills is usually exported in sacks.

The impurities of linseed consist, besides sand and earth, principally of the seeds of *Camelina dentata*, *Polygonum lapatlisfolium*, *Lollum arvense*, and *Spergula arvensis*. The presence of the first of these ("dotter") is regarded by buyers as a sign of genuine Riga seed. Good linseed (for sowing) should not contain more than 1 per cent. of foreign constituents. Thus, for example, if a sample of Russian seed were described as "7 or 7¼ measures," *i.e.* containing 7 or 7¼ parts of genuine linseed in 8 parts of seed (12·5 or 9 per cent. of impurities), it would be unsuitable. There are certain signs by which the fertility of linseed is judged, none of them reliable. The belief that seeds which float on water are not capable of germination, is as little true as the complementary belief that sinking in water indicates fertility. Another test is to press a handful of the seed between the fingers, when the good seeds will quickly slip out; but the good seeds themselves feel somewhat fatty and adhere to the hand. The following test is also much used:—A number of seeds are thrown on a red-hot metal plate or heated in a spoon over a flame; the seeds which burst are regarded as fresh and good, those which remain still as dead. This result is due to the larger or smaller quantity of water in the seeds, which, under the conditions mentioned, bursts the husk and by the recoil throws the seed out. The unreliability of this test has been shown experimentally; such methods of determining the fertility of seed have been replaced by a systematic test of fertility, carried out with a certain number of seeds chosen at random; in this manner alone can a reliable estimate be made. Good seed must have a fertility of 90 to 99 per cent.; in spite of the oil content, the seed retains its fertility longer than many other seeds. Seed two years old, which has been properly stored, may be used without hesitation, and is to be preferred to fresh seed of a lower quality.

The chemical composition of linseed is, according to Lousserpoult—

Oil	39 per cent.
Organic matter, free from nitrogen . .	19 ,,
,, ,, containing nitrogen .	20·5 ,,
Cellulose	3·2 ,,
Phosphates and other salts . . .	6 ,,
Water	12·3 ,,

The ash of linseed contains (Lichtenweiss)

Potash	25·9 per cent.
Soda	1·3 „
Lime	26·00 „
Magnesia	0·2 „
Ferric oxide	3·7 „
Phosphoric acid	40·10 „
Sulphuric acid	1·00 „
Chlorine	0·90 „
Silica	0·90 „

Into the Amsterdam linseed market come, in addition to native seed, most of which is used locally by small works and which always contains but little foreign seed, the different Russian, British-Indian, and La Plata varieties of seed. The last named is generally tolerably pure, and contains rarely more than 5 per cent. of impurities (foreign seeds, dirt, etc.), and the foreign seeds are almost exclusively non-oleaginous. Almost the same is true of the seed from British India; the impurities rarely exceed 5 per cent.; they consist principally of non-oleaginous seeds. At times *Brassica* seeds to the extent of 1 per cent. are found. From Calcutta comes a very poor quality of seed, known as "telqud," which often contains 30 per cent. and more of foreign seeds, which are frequently oil-bearing. The seed from Southern Russia (Black Sea) contains usually no more or little more impurity than the good seed mentioned above; occasionally bad samples occur. The foreign seeds always contain those of the *Brassica* species. The seed from Northern Russia (Baltic seed) is never pure. The amount of impurity varies; 5 per cent. is small, 10 to 20 per cent. is common, and higher percentages are found. The foreign seeds are to a considerable extent oil-bearing, consisting of seeds of the *Brassica* species, but chiefly of the dotter (*Camelina dentata*). North American seed is rare; it is always tolerably pure.

The foreign seeds are seldom oleaginous, and are therefore never estimated.

According to a private communication from a Dutch varnish maker, all varieties of linseed do not produce oils equally suitable for varnish making. The "hard" oils alone are suitable, whilst the "soft" oils are generally unuseable. In Holland the following varieties of seed are crushed:—

Archangel, which produces the "hardest" oil.
St. Petersburg, Riga, Steppes, Viatka, Samara, Siberia, Königsberg, Libau, which also produce "hard" oils, when the seed does not contain dotter.
Friesland, American, Azoff, Odessa, which produce less "hard" oils.
Bombay, Calcutta, which yield "soft" oils.

At the same time, it should be stated that it is very difficult to obtain the pure oils produced by the different varieties of seed, since the seed is mixed by the crushers before crushing, and also the oils produced are blended. The oils are examined by Fischer's hydrometer for oils; linseed oils of 29° to 30° are regarded as very hard, those of 30° to 31° as good, whilst linseed oil of more than 31° is not to be recommended.

Production of linseed oil.—The seeds are ground to a meal and pressed, with or without previous heating, or extracted by carbon bisulphide, carbon tetrachloride, petroleum spirit, etc.

Properties.—Cold-pressed linseed oil is very pale yellow; hot-pressed oil is more or less yellow or brown; extracted oil is also very pale yellow in colour. In taste linseed oil differs from the non-drying oils, it is peculiarly sweetish-bitter; the cold-pressed oil has a pleasant sweet taste like almonds; the hot-pressed oil has a burning after-taste. The odour is also peculiar and characteristic; with a little practice it is readily recognised.

Specific gravity at 15° C.	0·9347	(Schübler).
,, ,, ,,	0·9325	(Souchère).
,, ,, 18° C., raw oil	0·9299	(Stilurell).
,, ,, 12° C.	0·939	
,, ,, 25° C.	0·930	
,, ,, 50° C.	0·921	
,, ,, 94° C.	0·881	(Saussure).
,, ,, of the fatty acids at 100° C.	0·8599	(Archbutt and Allen).

Solidifying point of the oil: according to Gusserow, the oil solidifies after some days at −16° C.; according to Chateau, not below −27° C.; it melts at −16° C. to −20° C. (Glussner).

Melting point of the fatty acids	11°·12 C.	(Allen).
,, ,, ,,	17° C.	(Hübl).
Solidifying point of the fatty acids	13°·3 C.	(Hübl).
Saponification value	189–195	(Allen).
,, ,,	195·2	(Moore).
Iodine value	158	(Hübl).
,, ,,	155·2	(Moore).
Iodine value of the free fatty acids	155·2–155·9	(Morawski and Demski).

According to Mulder, the peculiar odour of linseed oil is not due alone to volatile fatty acids, butyric, valeric, capronic, etc. Linseed oil is soluble in 16 parts of ether and in 40 parts of alcohol at the ordinary temperature, and in 5 parts of boiling alcohol; with turpentine and petroleum spirit it is miscible in every proportion. Linseed oil boils at 230°, at 380° to 400° C. the malodorous whitish-grey vapour, already evolved at 300° C., takes fire spontaneously, and the oil then burns with a red flame, producing much smoke. When heated for several hours near the temperature at which it takes fire, linseed oil becomes a thick syrup; it then produces no grease mark on paper. Fresh linseed oil saponifies readily, with soda it forms a soft yellow soap, from the aqueous solution of which hydrochloric acid separates a limpid oil consisting of the fatty acids, which on cooling deposits crystals of palmitic and stearic acids. In the air it absorbs oxygen, and soon becomes rancid and viscous; in thin layers it dries to a neutral substance, insoluble in ether—linoxyn.

Pieszczek, of Königsberg, has made the following observations concerning a certain sample of linseed oil of poisonous action:—"Freshly pressed linseed oil is used in many districts, for example, in East Prussia, in the preparation of certain foods. From the producers of such an oil in this province I received a sample for examination, accompanied by the information that the oil 'caused general indisposition and dizziness' immediately after eating; in short, clear indications of poisoning. The material from which the oil was prepared was stated to be in 'good condition.' From its external properties the oil appeared to be of medium quality; there was nothing unusual in smell, taste, or colour. The specific gravity, iodine value, and saponification value lay within the permissible limits. A small percentage of oil emanating from plants belonging to the cruciferæ could be shown by means of the silver nitrate test, but this did not explain the toxic action. In order to probe the affair to the bottom I demanded an average sample of the seed from which the oil had been made. The examination of this sample gave a surprising result. The seed described as of 'good condition' contained 35 per cent. of impurities, which could be sorted out. Among the impurities were 15 per cent. of the seeds of *Lolium temulentum* (more properly, *L. remotum*). Thus there is no doubt that the toxic action of the apparently hot-pressed oil was due to this impurity, although by many *Lolium* is regarded as non-poisonous. The fact, well known to the farmer, that the horse avoids *Lolium*, may be equally due to the poisonous nature of this plant."

Hurst gives the following directions for testing the suitability of linseed oil for the practical purposes of boiling and paint making:—The specific gravity of the oil at 60° F. is first determined, which is best done by the specific gravity bottle. The bottle is weighed empty and dry, then filled with the oil, which is at 60° F., the formation of air-bubbles being avoided, the outside of the bottle is carefully cleaned, and it is then weighed. The weight of the oil, which is obtained by subtracting the weight of the bottle from the total weight, is divided by the weight of water which the bottle can contain. The result is the specific gravity, which, for pure linseed oil at 60° F., is 0·932 to 0·935. If it is impossible to work at 60° F., a correction must be applied. The number of degrees over or under 60° F. is multiplied by 0·00035, and the product added to or subtracted from the specific gravity obtained. For example, if 0·931 has been found at 65° F., the specific gravity is 0·93275 ($0{\cdot}00035 \times 5 = 0{\cdot}00175$). Oils which have a specific gravity below 0·930 or above 0·937 are to be regarded as unsatisfactory without further examination. The flash point of the oil is a further test. Linseed oil takes fire about 500° F.; if it contains mineral or rosin oil it takes fire at a lower temperature. An evaporating dish is placed upon a metal plate on a tripod, and protected from draughts. The dish is filled with the oil, and a thermometer with a sufficiently long range hung in it. The flame of a Bunsen burner is arranged below the plate carrying the dish, and both oil and thermometer observed. Vapours soon rise from the oil; the temperature is now observed, with a pure oil it should be 300° to 350° F. Then a small flame is moved just over the surface of the oil, until the vapours suddenly take fire with a blue flame. The temperature at which this occurs is the flash point; with pure linseed oil it lies about 500° F., rather above than below. If the sample contains mineral oil it flashes at about

400° F.; if it contains rosin oil, at about 320° F. Other adulterations are rare.

Preparation of linolic acid.—In order to obtain linolic acid, linseed or poppy oil is completely saponified with caustic soda. The soap is treated with hot water and caustic soda and purified by salting out repeatedly. It is then dissolved in much water and calcium chloride added, when the lime salts of the fatty acids are precipitated. The precipitate is washed with water, pressed, and treated with ether, in which calcium linolate alone dissolves, whilst the lime salts of the other fatty acids remain undissolved. The ethereal solution is decomposed with dilute hydrochloric acid, when calcium chloride goes over into the aqueous solution, the linolic acid remaining dissolved in the ether. The solution of linolic acid is separated from the watery solution, and the ether distilled off in a current of hydrogen at the lowest possible temperature. Dark yellow linolic acid remains, which, however, is not yet pure; it is dissolved in alcohol, and precipitated as barium linolate by means of ammonia and barium chloride. The barium salt is washed, pressed, and dissolved in ether, from which solution warty crystals separate. These are repeatedly recrystallised from ether. From the barium salt linolic acid is obtained by shaking with ether and dilute hydrochloric acid, removing the ethereal layer and distilling off the solvent in a current of hydrogen; the residual acid is dried under the air-pump over sulphuric acid and a mixture of lime and ferrous sulphate, which absorbs any oxygen present. Not quite pure linolic acid is obtained by decomposing lead linolate with sulphuretted hydrogen, and extracting with ether.

Linolic acid is a thin, slightly yellow oil of specific gravity 0·9215 at 15° C., strongly refractive, and with a weak acid reaction; the taste is at first mild, but afterwards harsh. It does not solidify at —18° C., is insoluble in water, but readily soluble in alcohol and ether. On standing in the air, linolic acid readily absorbs oxygen (about 2 per cent.), becoming viscous and sticky; it finally changes to a varnish-like mass. Thin layers upon wood dry to a varnish, but on glass become sticky. On treatment with hydriodic acid and red phosphorus, linolic acid is converted, by absorption of hydrogen, into stearic acid. It is not volatile without decomposition; on distillation other products are formed than from oleïc acid. It does not form sebacic acid. Nitrous acid turns linolic acid into a thick liquid; crystals of elaïdic acid or allied acids are not separated. Nitric acid produces a soft resin with considerable effervescence. The salts of linolic acid are obtained pure with difficulty, since they readily decompose with formation of acid salts. They are white and generally non-crystalline; on cooling of the hot alcoholic or ethereal solutions they separate in flocks; on spontaneous evaporation of these solutions a jelly is left. The salts of linolic acid become brown in the air, and acquire a peculiar smell. According to Hazura, the liquid fatty acids of linseed and poppyseed oils have the following percentage compositions:—

	Linseed Oil.	Poppyseed Oil.
Linolenic acid	15	5
Isolinolenic acid	65	...
Linolic acid	5	60
Oleïc acid	15	35

The linseed oil acids are used in varnish making to give elasticity to spirit varnishes, and very recently have been recommended as a solvent for copals. They have also been proposed as a priming for fresh cement, upon which oil paints do not stand, but in a short time soften and run off.

POPPYSEED OIL.

Mohnöl.

Huile de pavot somnifère, huile d'œillete, huile de pavot de pays.

This oil is obtained from the seeds of the poppy plant, *Papaver somniferum*, L., indigenous in the Eastern countries of the Mediterranean region. Since ancient times the plant has been cultivated in many parts of Europe, Asia, and Africa, and in more recent times in North America and Australia (New South Wales), partly as the material from which opium is obtained, and partly for the sake of its oleaginous seeds. There are two chief varieties of the poppy, *Papaver album*, D.C. and *Papaver nigrum*, D.C., the former of which bears white, the latter bluish-black or grey seeds. The white poppy produces the finest oil, and its seeds are those which are used for medicinal purposes. The oil is generally obtained from the black poppy, the cultivation of which has been found to be more profitable. Both varieties contain the same quantity of oil, 60 per cent. The smell and taste of poppy seed are well known, as also the form, which, accurately considered, is a flattened sphere, kidney-shaped in section. The weight of a poppy seed, according to Flückiger, when air dried, is about 0·5 mgrm. The surface of the seed shows ridges arranged in trellis fashion.

In each seed may be distinguished husk, embryo, and albumin (endosperm). The husk is about 0·014 mm. thick; it is enclosed by a skin covered with a thick cuticle, next to which comes parenchyma composed of collapsed cells. In the white-seeded variety this tissue has colourless contents, but in the dark-seeded variety, in the inner layer of the tissue are solid contents of brownish colour. The embryo is relatively large; the cotyledons and radicles are of equal length. The embryo is curved, it is composed of thin-walled cells, partly parenchymatous, partly composed of cambium tissue. The former contain oil drops in great quantity, and large aleurone grains, which in dimensions, form, and general structure agree with the analogous parts of the endosperm. This is composed of similar tissue, the cells of which are filled with small fat globules and large aleurone grains. In isolated aleurone grains is seen a clearer nucleus.

According to Sacc, white poppy seeds contain

	54·61	per cent. of	oil.
	23·26	„	proteïns.
about	12·00	„	albumins.
„	6·00	„	cellulose.

The seeds give 2 to 3 per cent. of ash, consisting chiefly of calcium phosphate.

Good poppy seed for sowing must contain kernels averaging at least 0·5 mgrm. in weight, must be uniform in colour, not contain more than 0·2 per cent. of foreign constituents, and must be fertile to the extent of 90 per cent. For the production of oil, complete ripeness and freshness are required; the freshness is recognised by the taste, and by an examination of the fertility. Direct adulteration with the seeds of wild varieties of the poppy cannot be successfully carried out, owing to the characteristic form, size, and colour of the garden poppy seed.

Production of poppyseed oil.—This is accomplished by crushing and pressing or extracting.

The production of poppyseed oil is an important industry in Northern France; the oil is used on the spot to the extent of about one-half, the remainder goes to Southern France, where it is used in the manufacture of special soaps. In Germany it is most largely produced in Baden, Bavaria, and Würtemberg. In the manufacture of the oil the poppy heads, as soon as they have reached a certain degree of dryness, are opened, their contents shaken out on an iron plate, fanned to remove portions of the head, and then ground in the mill to a meal, which is filled into a bag of ticking and pressed. The oil is caught in jars and casks, and allowed to stand until it is completely clear, when it is poured off and brought into the market.

In France two varieties of poppyseed oil are distinguished—white poppyseed oil, the food oil (*huile blanche*) from the finest seed and expressed at the first pressure, and the commercial oil, red poppyseed oil (*huile rousse*) obtained by the second pressing and from seed of poorer quality.

Properties.—The best poppyseed oil is white, and when fresh of pleasant taste; poorer qualities are golden yellow; oil of the second pressure is reddish.

Specific gravity at 15° C. .	0·924	(Souchère).
,, ,, ,, .	0·924–0·937	(Allen).
,, ,, ,, .	0·962	(Clarke).
,, ,, 18° C. .	0·9245	(Stilurell).
Solidifying point . . .	–18° C.	
Melting point of the fatty acids	20°·5 C.	
Solidifying point ,, ,,	16°·5 C.	(Hübl).
Hehner value . . .	95·38	(Dietzell and Kressner).
Saponification value . .	194·6	(Valenta).
,, ,, . .	192·8	(Moore).
Iodine value	136	(Hübl).
,, ,, ...	134	(Moore).

Poppyseed oil does not readily become rancid; it burns badly, it smokes less than olive oil and thus produces less soot, but it gives a bad light. It is soluble in 28 parts of cold and 6 parts of boiling alcohol, mixes with ether in every proportion; it is bleached by exposure to light. It possesses none of the action of opium. When subjected to dry distillation, poppyseed oil gives, on heating over an open flame without boiling, a thick colourless oily distillate, which partially solidifies on cooling, has a weak acid reaction, but gives up no trace of sebacic acid to water. When nothing more comes over, if the receiver is changed and more heat applied, the oil boils and produces much acroleïn, together with an oily distillate which imparts a strong acid reaction to water ; it contains sebacic and acrylic acids. If the distillation is finished when about half the oil remains, the retort contains on cooling a soft, thick substance, little coloured, with many of the properties of the anhydride of linolic acid, but more readily melted on heating.

Applications.—Alimentary: as olive oil and other fatty oils.

Technical: in the manufacture of soaps, as burning oil, and as binding medium in oil painting and the preparation of paints.

WALNUT OIL.

Nussöl.
Huile de noix.

Walnut oil is obtained from the well-known fruits of the walnut tree, *Juglans regia*, L., which belongs to the order of Juglandeæ; it grows in Northern Asia, Europe, and America. The fruit contains one kernel, surrounded by a tough, leathery, fleshy husk, which, in ripening, shrinks and becomes brown. The hard bivalved shell of the kernel encloses a bilobed swollen seed, oily and of pleasant taste. In regard to the form of the fruit, the following varieties are distinguished—the "horse nut," with a very large fruit of poor flavour; the "Meisennuss," with a very thin shell; the "Kriebelnuss," with a very hard shell; the "Schlägelnuss" (mallet nut), with an elongated kernel; the "butter nut," of long, round shape. The palatable kernel, when fresh, is surrounded by a thin bitter skin, which can be removed, but when the fruit is kept this skin loses its bitter taste and adheres fast to the kernel.

Production of walnut oil.—The fruits from which the oil is obtained must be 2 to 3 months old; when fresh they contain a whitish sap, which on pressing produces a turbid oil, brightening with difficulty. When kept too long the seeds produce a bad rancid oil, which also brightens slowly. The seeds contain 40 to 50 per cent. of oil; they are generally cleansed from the yellow seed skin, ground and pressed, first cold, when 30 to 35 per cent. of oil is obtained, then warm, when 10 to 15 per cent is expressed. The yield in hot pressing is about 45 per cent.

Properties.—The fresh cold-pressed oil is thin, almost colourless or pale greenish yellow; the colour, however, soon fades. The odour is pleasant, the flavour nutty; the oil readily turns rancid, and then possesses purgative properties. The warm-pressed oil is deeper in colour, and has a peculiar sharp smell and taste.

	Warm-pressed Oil.	Cold-pressed Oil.
Specific gravity at 10° C.	0·9300	0·9290
„ „ 12° C.	0·9290	0·9276
„ „ 15° C.	0·9286	0·9250
„ „ 20° C.	0·9236	0·9230
Viscosity at 15° C.	9·7 (water = 1).	
„ „ 7°·5 C.	11·8	

The oil is still thin at −15° C., at −17° to −18° C. it begins to thicken, forms a transparent mass at −24° C., and solidifies at −27° to −28° C. to a white hard fat. It dissolves in 100 parts of alcohol, and more readily in hot alcohol; on cooling, crystals of fatty acids separate. The oil contains the glycerides of linolic, myristic, and lauric acids.

Saponification value	196–197
Iodine value,	142–143
Melting point of the fatty acids	20° C.
Solidifying point „ „	16° C.

In regard to the iodine value, it should be observed that it rapidly falls with increasing oxidation of the oil.

Nitric acid gives a yellow coloration; nitric acid containing nitrous acid, a reddish yellow; fuming nitric acid, a dark red. Sulphuric acid of 1·7 specific gravity produces a brownish, then brown coloration. A mixture of sulphuric and nitric acids give first a brownish yellow, then a brown coloration. Zinc chloride is without action. Ammonia and caustic potash give yellowish-white emulsions; with caustic soda it is saponified just as linseed oil. Adulteration with bleached linseed oil is the most common; it is recognised by the soft resinous mass formed in the sulphuric acid test. Walnut oil possesses greater drying powers than linseed oil, and in drying does not crack.

Applications.—Alimentary: fresh cold-pressed oil is an excellent edible oil.

Technical: for oil paints and printing inks, in the manufacture of soap, and also, on account of the fine bright light, as an illuminating oil.

CASTOR OIL.

Ricinusöl.
Huile de ricin.

The common castor oil plant, *Ricinus communis*, L., and several closely allied forms, probably varieties of this species, but by many authors classified as distinct forms, produce seeds from which the oil is obtained in many warmer countries, where they principally grow. The habitat of *R. communis*, L., is India; *R. viridis* Willd., and *R. ruber*, Rumph., both of which are cultivated in the East Indies together with the first-named, also belong to the flora of India. In addition, *R. americanus*, *R. inermis*, Jacq., *R. lividus*, Willd., *R. africanus*, Willd., and probably other species also, are cultivated. The castor oil plant is most extensively grown in India, but also in certain West Indian islands; in North America, Algeria, and recently in Italy, there is a large annual crop of castor oil seeds, from which the oil is obtained. The castor oil plant was known as an oil producer in ancient Egypt; in the countries mentioned above, the plant began to be cultivated when the seeds were employed in the industry as a source of oil. Although the quantity of the oil used for medicinal purpose is at present considerable, it is unimportant as compared with the enormous amount used in the manufacture of soaps, for burning, in the preparation of Turkey red oil, as a leather oil, as a lubricant, and for other technical purposes.

The seeds of *R. communis* attain a length of 12 to 15 mm., and a width of 7 to 10 mm. The seeds from tropical countries are larger than the European. The seeds are oval, somewhat flattened on one side; on the other the projecting suture forms a longitudinal protuberance. The suture divides above into two, below it runs to the blackish, almost pentagonal hilum, which is frequently covered by a light-brown caruncle, bending forwards towards the suture. The husk of the seed is brown and speckled with grey. The seeds of *R. americanus* (from Martinique) are similar, but their husk is almost black, and the specks, which are comparatively few in number, are pale grey. The seeds of *R. inermis* (from India) are 12 mm. long, and 9 mm. wide, less flattened, with a scarcely protuberant suture, and a small greenish hilum; the husk is reddish brown, speckled with light brown spots. The seeds of *R. viridis* (from the Congo) are only 9 mm. long, and 6 mm. broad; the suture does not project, and the back is not flattened. The hilum is blackish and almost triangular in shape; the husk is greenish brown, with numerous light grey specks. The seeds are covered by a parchment-like skin, composed of flat polygonally grouped cells, which can be readily stripped off after softening in water; below this is a thin but very hard husk, composed of sclerenchymatous cells, to which is attached the soft skin of the seed. This is parenchymatous and traversed by many vascular bundles. In the interior of the seed lies the embryo, surrounded by much albumin. The cells, which are soft-walled parenchyma, contain fat globules and large aleurone grains, some of which enclose crystalloids. The seeds contain 40 to 45 per cent. of oil.

Castor oil is extracted in India in a somewhat primitive manner; in Madras three methods are used:—

(1) The seeds are roasted and then pounded in a mortar with three times the volume of water, the water is maintained at the boil and stirred with a wooden spoon. After some time the pot is removed from the fire, the oil skimmed off and allowed to cool. On the following day the mixture is again heated, and oil again obtained, which is of better quality than the first; it is separately collected and preserved.

(2) The castor oil seeds are first boiled, then allowed to dry in the sun for 2 to 3 days, pounded and treated as above in order to obtain the oil.

(3) The seeds are thrown into water over night, and ground in a mill to a paste, which is filled into a cloth and pressed. The oil produced is suitable for burning and industrial uses.

In order to decide which process gave the best result, official trials were made, quantities of 1 cwt. of seed being used in each trial.

(*a*) The seeds were ground in a crushing mill with horizontal rollers, the oily paste brought into a press and pressed.

(*b*) and (*c*). The seeds were roasted, as in the usual native process, pounded and boiled with water, the oil swimming on the surface being skimmed off.

The following yields were obtained:—

	a.	*b.*	*c.*
Oil	36·5 lbs.	32 lbs.	31·43 lbs.
Cake	36·8 ,,	not determined	43·48 ,,
Husk and impurities . .	27·7 ,,	,,	26·09 ,,

The yield (*c*) was obtained from seed ground in the ordinary native oil mill and then boiled. The oil (*a*) was very pure, the others were dirty and turbid. The quantity of oil obtained in (*a*), thus without the application of heat, is considerably larger than by the native processes, in which the yield is stated to be 25 per cent. of the weight of the seed.

In Bengal the seeds are only partially roasted in a pan, they are then pounded in a mortar without being previously husked. The paste is then mixed with water and placed on the fire in an earthen pan. The water should stand 6 to 9 cms. above the level of the pounded seed; as the water evaporates in boiling, the oil rises to the surface. The vessel is removed from the fire, allowed to cool, and exposed to the action of the sun. Through the warmth, oil again rises to the surface, it is skimmed off and heated until all the water has evaporated and the impurities have settled. According to another process, the seeds are boiled with water until they are soft, then dried and pounded; the yield is 32 per cent. from Chinalzis seed, 37·5 per cent. from Gohmna seed, and 31 per cent. from Tagur seed. The oil maker in Calcutta works in the following manner. The seeds are first cleaned by women, who are exclusively engaged in the operation. The seeds are spread out on a raised board, and broken in two or three pieces by a blow from a flat wooden hammer, the husks being loosened at the same time. The broken seeds are then separated from the husks by sieving, dried in the sun, powdered in a special apparatus, brought into bags and pressed. The oil as it flows out is caught in vessels placed below and brought into large galvanised iron tanks, in which it is exposed to the action of sunlight, in order to bleach it and separate the impurities. Oil which does not brighten is heated in a pan and, after treatment with charcoal, filtered through flannel. The oil obtained in this way is quite pure and suitable for medicinal purposes.

In another process, also used on the large scale, the preliminary treatment of the seed is the same as previously given, but in pressing the press is heated. This naturally gives rise to a larger yield of oil, but the oil acquires, through irregular heating, an unpleasant taste, detrimental to its quality. After pressing, the oil is heated to drive off moisture, and filtered through charcoal. Even in India the seeds are shelled by a machine, which consists of two smooth iron rollers moving towards one another, and turned by manual labour. The distance between the rollers can be adjusted so that seed of different sizes can be treated; the rollers must be so placed that the husk is split, as the seed passes through, without the kernel being injured. The seeds are sorted according to size (the rollers are adjusted to correspond with the dimensions of the seed) and placed in a wooden hopper, containing 4 to 5 kilos., above the rollers; they pass between the rollers, and then through a sieving arrangement by which the husks are separated from the kernels, which are next exposed to the sun. After this procedure, which is to be regarded as very important to the easy extraction of the oil, they are crushed (also between rollers) and converted into a coarse meal. The meal is at once filled into bags of coarse cloth, which are placed between the press-plates of an ordinary screw press, provided with two powerful screws with holes for a cross bar, each worked by two men.

The expressed oil flows into a vessel beneath, whilst a fire is maintained on the press. Each press-bag contains about 0·5 kilo. of ground kernels; the press takes 130 to 150 bags at once. The oil is thick, slimy, and contains water. It is heated in large copper pans in order to evaporate the water, of which it contains 10 to 20 per cent. The manipulation in this process of brightening the oil requires the greatest attention, for experience is necessary to hit the exact point at which the heating should be discontinued. The practised workman entrusted with the operation recognises the proper moment from the ceasing of the movements of the oil, and from the colour and appearance of the deposit which forms. Finally, after cooling the oil is filtered, when it is ready for market.

Properties of castor oil.—Castor oil is very viscid; it becomes still thicker on standing in the air, and is finally converted into a sticky mass. It has a mild flavour, with a biting after-taste; it mixes with alcohol in every proportion, and rotates the plane of polarisation of light, which is not the case with any other vegetable oil yet known. In thin layers it dries extremely slowly, forming a sticky skin, which even after months catches dust and fibres from the air; it does not in any way dry to a solid layer like linseed oil.

Specific gravity at 15° C. .	0·960–0·964	(Allen).
,, ,, ,, .	0·9613–0·9736	(Valenta).
,, ,, 18° C. .	0·9667	(Stilurell).
,, ,, 23° C. .	0·964	(Dietrich).
,, ,, 25° C. .	0·9575.	
,, ,, 94° C. .	0·9081	(Saussure).
Behaviour on cooling:—Solidifies at	– 17° to – 18° C.	
American oil solidifies at .	– 10° to – 12° C.	
Melting point of the fatty acids .	13° C.	
Solidifying ,, ,, .	3° C.	(Hübl).
Saponification value . .	181–181·5	(Valenta).
Iodine value	84·4	(Hübl).
Iodine value of the fatty acids .	86·6–88·3	(Morawski and Demski).

Composition.—The principal constituent of castor oil is the glyceride of ricinoleïc acid; it also contains stearin and palmitin. Pure castor oil is recognised by its property of mixing in every proportion with absolute alcohol and glacial acetic acid; it is soluble in 2 parts of 90 per cent., and 4 parts of 84 per cent. alcohol at 15° C. On the other hand, it is almost insoluble in paraffin oil, petroleum, and petroleum spirit; at 16° C. 0·5 per cent. of the oil is taken up by these solvents, at the same time the oil absorbs its own volume of petroleum ether, and 1½ volumes of paraffin oil or petroleum; the excess of solvent remains above the solution of solvent in oil. This behaviour, which is quite different from that of all other oils, makes its recognition in mixtures easy. The low saponification value, which approximates to that of the rape oils, is also characteristic. Castor oil gives the elaïdine reaction. The physiologically active constituent is more abundant in emulsions of the seed than in the oil. It has been found that castor oil produced by extraction has a stronger purgative action than expressed oil. Nothing is yet known as to the nature of this purgative constituent.

When air is blown into heated castor oil it is oxidised; it then acquires the property of mixing with mineral oils. This "soluble castor oil" is but little soluble in alcohol; it belongs to the oxidised or "blown oils." The behavior of castor oil towards sulphuric acid is important; it will be given under the heading of "Turkey red oil."

Recognition in other oils.—The presence of castor oil in other oil may be recognised, according to Draper, in the following manner. A few drops of the oil under examination are mixed with 5 to 6 drops of nitric acid. When the reaction is over the product is neutralised by sodium carbonate. As soon as the smell of nitrous acid has disappeared, the odour of œnanthylic acid appears, if castor oil be present. A parallel experiment with pure castor oil should be made for comparison.

Adulterations.—Castor oil is seldom adulterated; according to Schaedler, sesamé oil is used; the oil is then not completely soluble in alcohol, but gives turbid mixtures. Such an addition is also recognised by means of the elaïdine reaction, in which pure castor oil sets to a solid mass in 6 to 7 hours, whilst adulterated oil gives a sticky yellowish or reddish mass. In the reaction with a mixture of nitric and sulphuric acids, castor oil containing sesamé oil gives a bluish-green coloration.

Refining.—According to Pavesi, the oil is refined in the following manner:—1000 parts (by weight) are mixed with 25 parts of well purified bone black and 10 parts of calcined magnesia. The mixture is allowed to stand at 20° to 30° C. for 3 days with stirring, and is then filtered in a suitable manner. The filtered oil is colourless, and less inclined to thicken than unrefined oil.

Applications.—Medicinal: as a purgative.

Industrial: as a lubricant, in the manufacture of soaps, for Turkey red oil (used in dyeing), in varnishes to impart elasticity, etc.

Turkey red oil.—This substance, which is used in alizarin dyeing (in the production of reds upon fabrics), is obtained by the treatment of castor oil with strong sulphuric acid, as was stated by Müller-Jacobs in 1879. The product forms a thick, pale yellow, syrupy liquid of specific gravity 1·023, miscible with water in every proportion. By the action of concentrated sulphuric acid upon castor oil two substances are formed, one soluble, the other insoluble in water. According to Benedikt and Ulzer, the soluble portion is a sulphuric ester, ricinoleo-sulphuric acid, $C_{18}H_{33}O_2.O.SO_3H$, whilst the insoluble part is a mixture of free fatty acids and unsaponified oil. Juillard, on the other hand, considers that Turkey red oil is of a more complicated composition, and not always uniform. It contains the sulphuric acid esters of ricinoleïc acid, several polyricinoleïc acids, and compounds of triricinoleïn, mixed with their decomposition products, among which ricinoleïc acid predominates. In the action of sulphuric acid upon castor oil, the glycerin is not completely split off, but half remains behind in the oil, thus influencing its solubility and capability of forming emulsions. On boiling with alkali, Turkey red oil is decomposed into ricinoleïc and ricinoleo-sulphuric acids. With water or acids, on the contrary, pentaricinic, triricinic, $HO.C_{17}H_{32}.CO_2.C_{17}H_{32}CO_2.C_{17}H_{32}.CO_2H$, and diricinic acid, $HO.C_{17}H_{32}.CO_2.C_{17}H_{32}.CO_2H$, are formed. By the action of sulphuric acid upon castor oil at a low temperature triricinoleïn-sulphuric acid is first formed, which is hydrolysed, monoricinoleïn-sulphuric and ricinoleïc acids, also polyacids, being formed.

According to Scheurer-Kestner, the acid product of the reaction is not a solution of fatty acids in water, but a hydrate containing 10 mols. of water to 1 mol. of oil.

Turkey red oil contains two constituents which may be separated by treating with ether; the insoluble oil is lighter than water, difficult to saponify, has an acid reaction, and dissolves in alkalis without decomposition. The soluble oil is heavier than water, in which it forms a clear solution; it contains 8·8 per cent. of sulphuric acid. When salted out by Glauber's salt it contains 30 to 40 per cent. of water, which is partially lost at 75° C.

Another preparation, also obtained by the action of sulphuric acid on fats, may be used in place of Turkey red oil. Schmidt and Tönges, by acting upon fats or fatty acids with strong sulphuric acid, obtained the glycerides of the sulpho-fatty acids or the sulpho-fatty acids themselves respectively. These substances, when heated to 105° to 120° C., produce the glycerides of the hydroxy-fatty acids or the acids themselves. They can also be converted into dihydroxy-fatty acids by again treating with sulphuric acid, and heating the sulphuric acid esters produced.

The following methods have been given for the preparation of Turkey red oil:—

(1) Not more than 20 kilos. of castor oil are weighed off in a thin-walled earthenware or enamelled iron vessel, 2½ kilos. (or ⅛ the weight of the oil) of strong sulphuric acid are gradually added in a thin stream. Never more than 100 grms. of the acid are added at once. During the operation the mixture must be continuously stirred, otherwise the acid sinks to the bottom; the oil is coloured brown, and sulphur dioxide is evolved. The vessel is kept at 30° to 40° C. for 4 hours on the water-bath, and then left at rest for 24 hours. The contents of several vessels are poured into a wooden tub, and excess of acid removed by three washings with a 10 per cent. solution of common salt, 4 litres of which are used to 5 kilos. of oil. The first two washes are done in the cold, the third warm. After adding the salt solution the mixture is well stirred and left to stand 6 hours, when the mixture of salt solution and sulphuric acid is drawn off from the bottom. The oily product has a strong acid reaction; it is seldom used in this condition, since it gives a turbid solution in water. The oil is neutralised by the addition of 10 per cent. soda solution, or caustic soda solution of 5° B. and ammonia of specific gravity 0·961. To each 5 kilos. of oil 2 litres of soda solution, or 0·5 to 1·5 litres of caustic soda and 0·5 to 1·25 litres of ammonia are used. The oil must now have a light brown colour and be completely clear. An excess of soda should be avoided, since this hinders the complete absorption of the oil by the fibre. Excess of ammonia is not detrimental, but it must be added last. The oil so obtained gives a clear solution in water in every proportion; if the solution should be turbid, it can be cleared by the addition of a little ammonia.

(2) Castor oil is treated by 20 per cent. of sulphuric acid of 66° B., by allowing the acid to flow slowly into the oil in a thin stream whilst stirring. The oil is contained in a lead-lined iron vessel, cooled by ice. After standing 2 to 3 hours the mass is gradually diluted under steady stirring by luke-warm soda solution, 2·8 kilos. of crystallised soda being used to 1 kilo. of acid. After standing over night the finished product separates. Liechti uses in winter 20 to 30 per cent., in summer 15 to 20 per cent. of sulphuric acid without cooling, but warming in winter to a summer temperature.

Turkey red oil apparatus of Schöhfleuthner.— In preparing castor oil soluble in water, an oaken vessel, about 1·2 m. wide and 0·3 m. high, lined with lead, is used. It is filled to a depth of 5 cms. by about 60 litres of oil; the contents can then be rapidly mixed, which is important, since the rapid development of heat on the addition of sulphuric acid may cause the failure of the operation. When brought in contact with 0·9 parts of sulphuric acid, the oil is quickly destroyed, thus great care and attention are required to bring the process, which lasts several hours, to a proper termination. The apparatus of Schöhfleuthner is very convenient for this purpose; the acid is contained in bottles provided with taps, and fastened in wire baskets to the arms of the stirring apparatus. The bottles rotate with the stirrer, and are gradually emptied into the vessel. The arms of the stirrer consist of rods, 6 × 6 cms., well coated with lead, and carrying narrow pointed rods arranged one above the other in the form of a grating. The stirrer, by means of suitable mechanism, can at once be raised out of the liquid, as is necessary at the end of the operation, or at other times. The Turkey red oil made by this method is dark orange to pale red in colour, is clear, and when carefully washed forms no precipitate.

Preparation of a substance from castor oil miscible with mineral oils and water.—Castor oil is insoluble in mineral oils, and thus cannot be used in the preparation of mineral lubricating oils. When castor oil is heated and subjected to a partial dry distillation, a semi-solid sticky residue remains, which mixes even at the ordinary temperature with mineral oil in every proportion, and can also take up certain quantities of cerasine and vaseline ; the new product can also take up water and aqueous solutions, even together with mineral oil. These properties make the oily product particularly suited to the preparation of viscous lubricating oils in admixture with mineral oils, and also as a base for pharmaceutical salves and liniments.

SUNFLOWER OIL.

Sonnenblumenöl.
Huile de tournesol.

The sunflower (*Helianthus annuus*, L.), from the seeds of which the oil is obtained, is a native of Mexico. The plant has long been grown in European gardens for decorative purposes, and also is cultivated on a large scale in Southern Russia. In its native country oil was long ago extracted from sunflower seeds, and in the previous century attempts were made in Germany to make the plant useful as a producer of an industrial oil, but unsuccessfully. The seed kernels are used in Russia for the production of oil, and are also exported to other European countries for the same purpose. Russian sunflower oil is a not inconsiderable article of commerce in Germany.

The fruits of the sunflower are either black, greyish brown, or whitish; in commerce, black fruits or black with brown longitudinal stripes are alone found. When dry the fruits form longish ovals, somewhat sharpened, somewhat sunken or at least flattened at the broad (upper) end, about 1 cm. long, 4 to 5 mm. wide, and 3 to 4 mm. thick. On careful examination the fruit is found to be unsymmetrica The woody brittle husk, not readily split along the length, i 0·5 to 0·6 mm. thick. The external surface, which is somewhat polished, is either quite black or has a black ground with longitudinal greyish-brown stripes. The weight of the husk is in proportion to the weight of the seed as 53 to 47, both air-dried. Since the dried fruit produces 15 per cent. of oil, the seed must contain about 32 per cent. The fruit capsule is covered by a membrane which consists of long flattened cells with transparent walls thickened towards the exterior, and with brownish-black resinous contents. To this membrane is attached a second consisting of parenchymatous tissue containing one or more layers of cells. Next comes the tissue which constitutes the principal part of the capsule; it consists of long thick-walled parenchyma cells traversed by numerous vessels and intersected in a vertical direction by a tissue, which, in arrangement, shape, and position of the cells, can best be compared with medullary rays. A section of the external skin of the capsule appears to be divided into flat sheets at distances of 0·20 to 0·38 mm., from the junctions of which the medullary tissue issues. The skin of the seed, as seen from the surface, consists of several layers of cells polygonally grouped; it is traversed by vascular bundles which consist either of cambium elements or principally of thick spiral vessels. In the seed itself is visible only parenchyma and the epithelium covering the cotyledons; the latter differs from the former only by its flattened and light brown cells, the parenchyma cells being rounded or polyhedral. Both in the cells of the epithelium and of the parenchyma fat drops occur, and also aleurone granules crowded together, 0·0036 to 0·0067 mm. in diameter; when these are examined immersed in oil by the highest powers of the microscope, grains of varying sizes are seen in the interior.

Production of sunflower oil.—The seeds are shelled by gentle crushing, the kernels ground, and the oil expressed or extracted.

Properties.—Sunflower oil is clear, pale yellow, has a pleasant smell and a mild taste; it has not yet been thoroughly examined.

Specific gravity at 15° C. .	0·962 (Chateau).
" " " .	0·924–0·926 (Allen).
Melting point of the fatty acids	23° C.
Solidifying point of the oil .	− 17° (Bach).

Applications.—As an edible oil, like olive and other vegetable oils.

Technical: in the manufacture of soaps, and in paints instead of linseed oil.

GRAPESEED OIL.

Traubenkernöl.
Huile des pepins des raisins.

The material from which this oil is obtained is the seed of the grape, which together with skins constitutes the residue (" trester ") after the sweet juice has been expressed. The plant itself, *Vitis vinifera,* is too well known to need further description. The seeds from which oil is to be obtained must be entirely freed from the skins and pulp, since these find another use. When the " trester " is to be worked for brandy the seeds must be picked out by children, because it produces a much better product when free from seeds. In all other cases the " trester," as it comes from the press, is spread out on a large drying floor and turned over every day with a fork; it soon shrinks so that the stalks can be removed by a fork. When the skins have still further dried, the seeds can be separated from them by a fan; seeds which still remain may easily be loosened by thrashing. The seeds so obtained are spread out in a thin layer upon the floor, with a free access of air, and well dried, which is very important for the production of good oil. Statements as to the amount of oil in grape seeds vary greatly; it is said that 12 or even 20 per cent. of oil can be expressed.

Production of grapeseed oil.—The dried seeds are either ground fine in an ordinary mill with horizontal stones, or in an oil mill with vertical runners. During the grinding, lukewarm water is added from time to time to prevent adhesion to the stones. The ground seed, which produces the more oil the finer it is ground, is brought into a copper vessel and gradually mixed with ¼ to ⅓ of its weight of warm water, the formation of lumps being prevented by stirring. The mass must not be allowed to burn on the pan sides, otherwise the oil acquires a burnt taste. The mixture is now brought into ordinary hair press bags and pressed in an oil press. When no more oil flows out, the cakes are again several times ground and treated in the manner previously described, when they give up more oil. In this manner, from 100 kilos. of seed, 10, 12, and even 20 kilos. of oil are obtained. These differences in the yield appear to be due to differences in the variety of grapes, and in the soil upon which the plant grows.

Properties.—Grapeseed oil is a somewhat viscous golden yellow or brownish-yellow oil, inclining to green. The odour is slight and characteristic; the cold-pressed oil has a mild flavour, and is a valuable edible oil; when hot pressed it has a somewhat harsh taste.

The specific gravity of the oil at 15° C. is 0·9202; it solidifies to a butter at —11° C. In the air it turns rancid, becomes brown, and dries slowly. It burns with a bright flame, free from smoke and smell. It can be refined like all fatty oils.

R. Wagner found in grape seeds from Lower Franconia, dried at 100° C., 11·2 per cent. of oil, and in another experiment 10·8 per cent. Fitz found in grape seeds 15 to 18 per cent. of oil, which contained the glycerides of palmitic, stearic, and erucic acids, the latter forming about one half of the fatty acids.

Applications.—As an edible oil, like olive and other vegetable oils.

Industrial: as a lubricant, for soaps, and as a burning oil, etc.

New Drying Oils

I'Sano or **Ungucko oil.**—The seeds are obtained from a large tree of the family of the *Oleaceæ*, which grows exclusively in Kuabi, near Brazzaville, and in the Loango and M. Pongu districts of the French Congo. The fruit is a bean about 3 cm. long, containing a brown oval nut 2·7 cm. long and 2·4 cm. thick. The shell is about 1 mm. thick. The seed almost entirely fills the cavity of the shell, to which it is attached at the bottom; in taste it resembles the hazel nut. The fruit is much liked by animals, although the natives do not use it as food. The kernel, which is 63 per cent. of the weight of the whole fruit, contains sap; on extraction by benzene it yields 60 per cent. of oil. This oil has a reddish colour, an acorn-like flavour, and peculiar smell. It is very viscid, dries well, is slightly soluble in 90 per cent. alcohol, and has the specific gravity 0·973 at 23° C. By treatment with boiling water and litharge it is converted into a paste, which hardens after some time. On account of its composition the oil is suited for industrial purposes, and may probably be used instead of linseed oil, with which it has great similarity.

Mohamba oil.—The seeds are in many respects similar to the I'Sano; the kernel constitutes 65 per cent of the fruit. The husk, as also that of I'Sano, contains insufficient nutritive material to possess commercial value. By extraction with benzene the kernels give 12 per cent. of oil, and leave a residue containing 19·5 per cent. of nitrogenous matter (3·42 per cent. of nitrogen), 7·62 per cent. of which is soluble in water, 1·5 per cent. of sugar, 4·5 per cent. of gum, and 43·33 per cent. of gum, starch, etc., insoluble in water. The ash is similar in composition to that of I'Sano cake. The cakes form a nutritious food rich in nitrogen and starch. Mohamba oil is fluid at 15° C.; its colour is yellow, and taste unpleasant; it is without odour. The specific gravity at 23° C. is 0·915, and the solubility in 90 per cent. alcohol very small. When treated with boiling water and litharge it forms a solid plaster.

DRYING OILS OF NO TECHNICAL IMPORTANCE

The drying oils mentioned below have no importance in commerce, either because they are introduced in too small quantities and at irregular intervals, or because their usefulness has not been examined, so that no definite statement can be made regarding it. It is probable that none of these oils, especially such as come from distant countries, have yet been brought at any time into the market in large quantity. This was the case for many years with Chinese and Japanese wood oils, which in a short time have found tolerably extended applications, thanks to their remarkable properties.

Shial Kaata oil (Argemoneöl, Huile de pavot epineux, huile de charbon jaune). From *Argemone mexicana.*

Deadly nightshade oil (Belladonna seed oil, Belladonnaöl, Tallkirschensamenöl, Huile de belladonna). From *Atropa belladonna.*

Camul oil (Camulöl, Huile de Polongo). From *Mallolotus Philippinensis*, Mull. (Abyssinia, Southern Arabia, India, Malay Archipelago, Philippines, East China, North Australia).

Koeme oil (Castanhasöl, Huile de noix d'Inhambane). From *Pelfairia pedata*, Hook.

Chironji oil. From *Buchanania latifolia.*

Colocynth seed oil. From *Cucumis colocynthis.*

Coula-nut oil (Coulanussöl, Huile de noix de Coula). From *Coula edulis* (West Coast of Africa). This oil does not dry completely.

Thistle oil (Distelöl, Huile de chardon). From *Onopodon acanthium*, L.

Red pineseed oil (Fichtensamenöl, Huile de pinastre). From *Picea vulgaris*, Lank.

Garden cress oil (Gartenkressensamenöl, Huile de cresson alenois). From *Lepidium sativum*, L.

Horned poppyseed oil (Hornmohnöl, Huile de pavot cornu). From *Glaucinum luteum*, Scop.

Cucumber seed oil (Gurkenkernöl, Huile de concombre, Huile d'Egusi, Huile d'Abobora). From *Cucumis sativus*, L.

Hickory oil (Hickoryöl, Huile de hickory). From *Carya alba.*

Jy-chee oil. From *Euphorbia dracunculoides*, Lam. (Punjab, Madras, Bengal). A drying oil for paints.

Pine oil, Scotch fir oil (Kiefernsamenöl Föhrensamenöl, Huile de pin). From *Pinus sylvestris*, L.

Burr oil (Klettenöl, Huile de Bardanne). From *Arctinum Lappa*, L.

Pumpkinseed oil (Kürbiskernöl, Huile de pepins de citrouille).

Melonseed oil (Melonöl, Huile de melon). From *Cucumis melo*, L.

Weldseed oil (Resedasamenöl, Huile de gaude). From *Reseda luteola.*

Honesty oil, Hesperis oil (Rothrepsöl, Huile de julienne). From *Hesperis matronalis.*

Safflower oil (Saffloröl, Huile de carthame, huile de saffre). From *Carthamus tinctorius*, L.

Tobaccoseed oil (Tabaksamenöl, Huile de tabac). From *Nicotiana tabacum.*

Spruce firseed oil (Tannensamenöl, Huile de sapin). From *Abies pectinata*, D.C.

Cassweed seed oil (Taschelkrautsamenöl, Huile de thlaspi, Huile de cresson). From *Thlaspi arvense*, L.

Water melon oil (Wassermelonöl, Huile de melon d'eau, Huile de Beraf). From *Curcubita citrullus.*

Zachun oil (Zachunöl). From *Bolonites Roxburghii Planch.* (Africa, Senegal).

Cembra-pineoil (Zirbelnussöl). From *Pinus cembra.*

CHAPTER III

PRODUCTION OF THE DRYING OILS BY EXPRESSION AND EXTRACTION. REFINING AND BLEACHING

Drying oils are produced from the under-mentioned seeds and fruits, which contain the proportions of oils stated:—

	Per cent.
Candle nut, from *Aleurites moluccana*	62–64
Cedar nut	
Hempseed, from *Cannabis sativa*	30–35
Linseed, from *Linum ussitatissimum*	38–40
Pumpkin seed, from *Cucurbita pepo*	20–25
Poppy seed, from *Papaver somniferum*	41–50
Walnut, from *Juglans regia*	63–65
Seeds of *Elæococca vernicia* (Wood oil)	
Castor oil seed, from *Ricinus communis*	46–53
Sunflower seed, from *Helianthus annuus*	21–22
Grapeseed, from *Vitis vinifera*	11–12

The above percentages of oil cannot be obtained from the raw materials; the yield is always smaller by 8 to 15 per cent. by expression, and by 4 to 8 per cent. in extraction. The seeds themselves contain very varying proportions of oil, according to the locality and climate. It is thus frequently necessary to estimate the oil in particular parcels of seed; this is best done by extracting the oil, by means of a solvent, from the powdered and dried substance, under conditions which permit of complete extraction. Vohl's oleometer is a suitable apparatus for this purpose; the whole apparatus, as shown in

sts of four parts, the
nd the condenser D.
the narrower tube *b*
with B. The tube *c*
, in which the tube *o*
to the bottom of the
sed by the liquid in
c has another side
is the tube *g* drawn
head C is connected
ure *f* by means of *i*,
be *c* of the extractor.
by *k* with the tube *l*
n, *l* is constricted to
iameter. The appar-
owing manner:—The
sed at *d* by a plug of
hen affixed by means
and ground seed is
h the tubulure *f*; it
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d; it is convenient to
When so arranged the
apours rise through *c*
ack into B, until the
perature at which the
the case the vapours

rise through *h* into C, where they at first condense; but afterwards, when C has become hot, they reach *l*, where they are condensed by the cold water or ice, the liquid flowing back by the bent tube *k* into C, thence through *i* and *f* into A, and finally through *c* into B. The tube *g* serves for the exit and entry of air during changes of temperature. In this manner it is possible to extract considerable quantities of seed by relatively little petroleum spirit. When the liquid runs from *c* clear and colourless it may be assumed that the oil is completely extracted. The petroleum spirit is now separated by distillation from the oil and the latter weighed. The petroleum spirit (canadol) used for this process should not exceed 0·66 to 0·68 in specific gravity, or 50° to 60° C. in boiling point. With some practice such an estimation may be made in 1½ to 2 hours. When many similar estimations have to be made, the final removal of the spirit by distillation takes too much time, requires great care, and, in addition, the necessary apparatus, which is not always at hand. For these reasons Vohl has endeavoured to estimate the oil content of seeds volumetrically. The specific gravity of the fatty oils is considerably higher than that of the petroleum spirit by means of which they are extracted, thus the specific gravity of the spirit solution must rise in proportion to the increase of the quantity of oil it contains.

Before proceeding to consider the actual production of the oils, it is necessary to make a few remarks concerning the storage of the seeds and fruits, since the quality of the oil depends on the faultless nature of the raw material, and in the storage no deterioration must be allowed. The seed also requires purification; all oil seeds, as they come into the market, contain foreign admixtures, earth, sand, the seeds of weeds, and oil-bearing seeds, all of which should be removed as far as possible. In producing oils of this category, seeds containing non-drying oils must not be present, since they f the oil and cause

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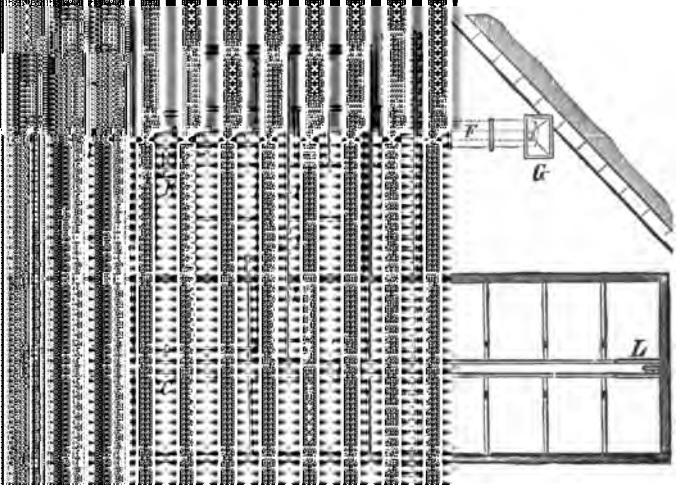

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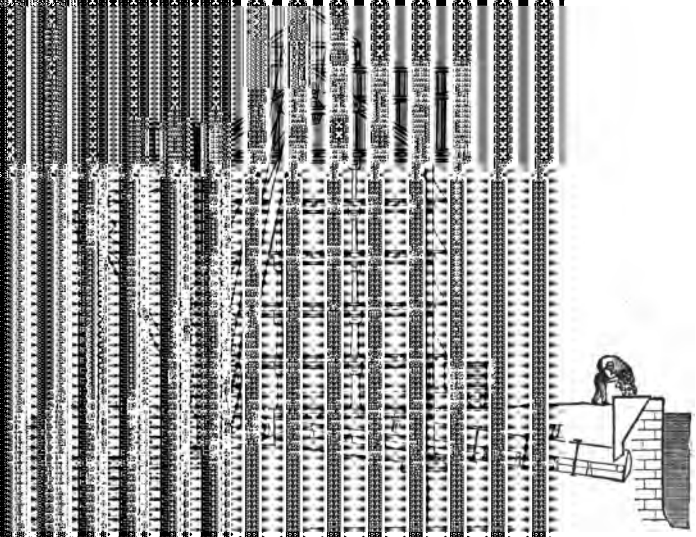

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[illegible] medean screw, [illegible] or of the store, [illegible] raised by the [illegible] ated iron roof [illegible] it is a con- [illegible] ly; they fall [illegible] distribution is [illegible] floors are con- [illegible] be conveyed to the lowest room. In moving the seed the valves of 150 pipes, each 5 cm. wide, communicating from floor to floor, are opened, the seed then falls uniformly down until it attains a depth of 1·5 metres, each seed acquiring a rotary motion. The seeds collect in conical heaps. All the valves are connected by levers; the whole management and labour of the store lies in the hands of one workman. In order to raise the seeds again from the bottom to the top floor, they are brought into the three Archimedean screws, which traverse the length of the store; these feed a cross screw which leads the seeds into the hopper K of the elevator B. The series of operations can then be recommenced.

Circumstances may make desirable a different direction of movement, on this account the pillars are traversed by pipes with lateral feeders through which the seeds may enter. By this means the seed may be transferred from the top floor direct to the bottom, omitting the intermediate floors. For example, if new seed is to be admitted, the particular floor may be cut off without interrupting the regular work of the store. The ceilings of the store are of corrugated iron. Each square metre of floor space can be loaded with 1500 kilos. of seed. The columns are braced together by ties to support the horizontal thrust of the corrugated iron. The conveyers and elevators are run from the shafting W, connected with the general system of the oil mill. Wherever the belts have to run through the unprotected seed, iron casing of the necessary height is provided. Particular attention has been given to the ventilation of the store; the shutters are entirely of iron, and can be opened or closed by rods which connect a number of windows. A short elevator can also be provided, traversing only three storeys; this is of particular advantage when the store is connected with other warehouses. Arrangements must then be made to convey the seed in the horizontal direction, for when it is to be moved from one store to the other a screw conveyer is not suitable, especially when the distance is great. Another conveyer has recently come into use, it consists of a broad indiarubber or cotton band running in a horizontal direction, and always kept stretched. The seed is deposited on one end of this belt, which is 300 mm. wide, and is carried forward with it, without a single seed escaping over the edge, until the direction of movement becomes vertical. This method of conveying seed tends to preserve and purify it.

Cleaning the seed.—All oil seeds, before they are used, must be cleansed from impurities, such as sand, earth, portions of plants, and foreign seeds, which naturally accompany the seed. Otherwise the cake will vary in flavour, and the sand and earth will not only injure the machinery but also deteriorate the quality of the cake, since it will contain substances of no nutritive value. Those impurities which are lighter than the seed can be removed by fanning, the current of air carrying them away in consequence of their lightness. The foreign seeds, which might also injure the quality of the oil, fall into the following classes:—

1. Seeds, which themselves produce oil, alter the yield of oil very little, but injure its quality when, for example, the seeds of non-drying oils accompany those producing drying oils. Thus in linseed there are generally found the seeds of charlock, mustard, and dotter, since these usually accompany the plant as weeds.

2. Seeds, which produce no oil when pressed, but contain so much oil that they absorb none from the meal but increase its volume, for example, the seeds of fleawort and corn spurry.

3. Seeds which contain no oil or very little. These absorb oil when the meal is pressed and thus reduce the yield. Such are the seeds of flaxweed and darnel. The earthy and fortuitous admixtures also absorb oil, diminish the yield, and make the cakes heavier.

In the further treatment of the seed the size of the impurities is of great importance, whether they are equal in size to the oil seeds, greater or smaller. If they are equal in size it is difficult to remove them, since they pass through the sieve together with the seed, if they are not sufficiently friable (as earth, etc.) to be broken up in the sieve. The separation of impurities larger or smaller than the oil seeds is easy; they are removed by sieving machines in which the seed is shaken.

PRODUCTION OF THE DRYING OILS BY EXPRESSION AND EXTRACTION

The drying oils are obtained, like all seed oils, by (1) cold or hot pressing, or (2) extraction by a volatile solvent, such as carbon bisulphide, petroleum spirit, canadol, carbon tetrachloride, etc., which can be completely, or almost completely, removed from the seed after the extraction of the oil. Cold pressing produces pale pure oils free from foreign admixtures, and therefore of good flavour, which in many districts are used for culinary purposes. At the same time, the yield is smaller and the cakes contain more oil; they are not generally used as fodder, but are ground and subjected to hot pressing, or are extracted, the latter being more profitable in regard to the yield of oil. Hot pressing, which may be accomplished by means of heated presses or by previously heating the seed, gives rise to a more coloured, turbid, impure oil, unsuited to culinary uses. By extraction a paler and more pure oil is obtained, but equally unsuited for edible use. For technical purposes the seeds and fruits which yield drying oils are exclusively hot pressed or extracted, because the yield is all important and the oils produced completely satisfy commercial requirements.

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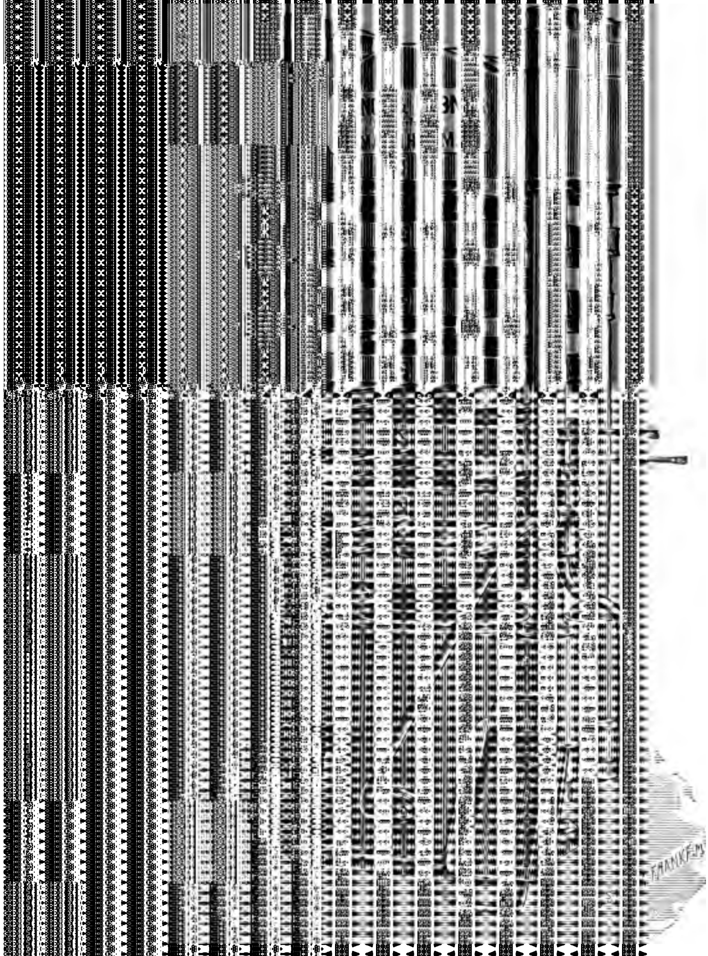

[illegible] Hydraulic Presses

[illegible] seeds, and, in [illegible] ic [illegible] use, are now [illegible] ses are almost exclusively employed. These may be of the pan, trough, sack, ring, or horizontal type, and may be with or without press cloths, according to the construction of the press. Of the different systems, that is naturally the most efficient which is of the simplest construction, can be worked rapidly and easily, is of sufficient power to express the oil, is durable, requires the least repairing, and produces uniform cakes without the use of press cloths. Such a press is illustrated in Fig. 4, the construction is broadly as follows:—In the press 4, 6, 8, or 10 wrought-iron or steel rings are built above one another, each of which has a moveable bottom of finely perforated steel. Between every two rings is a cast-iron or cast-steel plate, the upper surface of which is grooved, the lower plane. On these plates, which are introduced between the columns of the press, are slides in which the press rings move, and which serve as guides in the withdrawal and introduction of the rings. Each plate has a large peripheral channel to collect the expressed oil. The press is filled as follows:—Upon the perforated steel plate of each ring is laid a cover of woven horsehair, wool, or felt, upon which is placed the seed, and then another horsehair cloth. When all the rings are filled the pressure is applied, forcing the upper grooved surface of each plate into the ring above it; the oil flows through the horsehair cloth, the perforated steel plate, and the grooves of the plate, into the channel. If the oil flows uniformly from the whole surface of the cake it has only to traverse the thin cake; it therefore runs off readily and rapidly, and cakes of accurate shape and uniform composition are produced. The edges of the cake, which do not contain more oil than the centre, need not be trimmed off. In presses for very large cakes the rings are too heavy to be lifted out; they are therefore provided with systems of slides and a double set of rings. After one set of rings has been pressed it is withdrawn to one side for emptying and filling, whilst the other full set is at once pushed into the press from the other side. The method of working is continuous and very simple.

By using press boxes of corresponding shape in place of the rings, circular, rectangular, or trapezoidal cakes of any dimensions can be produced. All the presses are also provided with heating arrangements for steam or hot water, by which each plate is heated. Since each cake is placed between two heated press plates, the heating is uniform and thorough. These presses produce results unattainable by other systems. In order to be able to bring into each ring the largest possible quantity of seed, a special filling machine is provided which fills in one minute without attention eight rings, and compresses the charge so that each ring contains about 20 per cent. more seed than if it were filled by hand. These machines may also be supplied with heating pans. One machine is sufficient to supply fifteen presses; they are to be recommended for large works. All the presses are constructed for a working pressure of more than 300 atmospheres; pressure cylinders, columns, and nuts are of cast steel.

Accumulators are a great advantage in the uniform working of hydraulic presses; they are in general use.

The size of the press, the form and dimensions of the press troughs, boxes, and rings naturally vary with the amount of drying oil which is to be produced.

Before the oleaginous raw material can be treated by the presses, it must be crushed or ground and then heated. In the older oil works ordinary mill-stones or stamps were generally used for grinding, then edge-runners were employed, whilst now roller crushers are in almost exclusive use. The machines have at least two, and frequently four to six, rollers, placed one above the other, between which the seed passes and is crushed. The rollers are generally arranged so that the seed is completely ground in its descent between them, and then falls as meal into a receiver below. It is absolutely necessary, in the first place, to pass the material to be ground through some efficient sieving arrangement, in order to remove from the seed all hard substances, such as stones, etc., which might injure the rollers and make re-turning necessary before they could again be used. The grinding machinery is also used to regrind the cakes obtained in the first pressing, to prepare for the second pressing, so that the presses may again be charged with the material. Disintegrators are much used for powdering oil cakes; they have an enormous capacity and turn out very fine meal.

The meal obtained by one of the ordinary methods is pressed in cold presses if it is desired to produce edible linseed, poppy, or walnut oil; in the case of oils for industrial purposes (which predominate), the meal is previously heated in order that the oil may run out more easily in pressing. This heating was formerly done in a very primitive manner in iron pans, heated over an open fire or by hot water; the meal was liable to accidents, especially to overheating, which not rarely caused the production of strongly smelling and burnt oils.

The arrangements now used for heating the seed meal may be very simple or very intricate; in the most recent installations the heating is frequently combined with arrangements for filling the press bags or plates and for moulding the cakes. The size of the pan depends on the capacity of the presses, it should not hold more meal than is required for one charge of the presses.

The moulding machine is designed to compress the ground and heated material, which is in a loose condition, into a smaller volume, but not beyond the point at which oil just begins to flow out. For this purpose great pressure is not required; the presses are therefore driven by steam. By charging the oil presses with cakes which have received a

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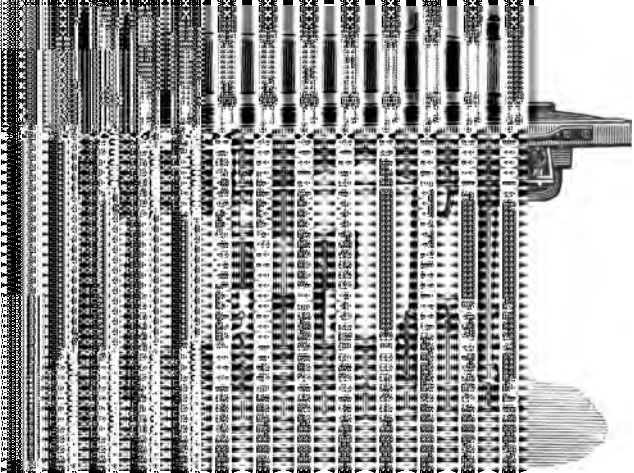

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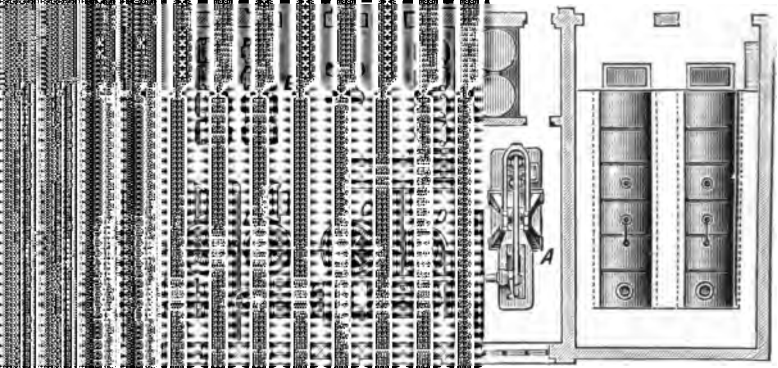

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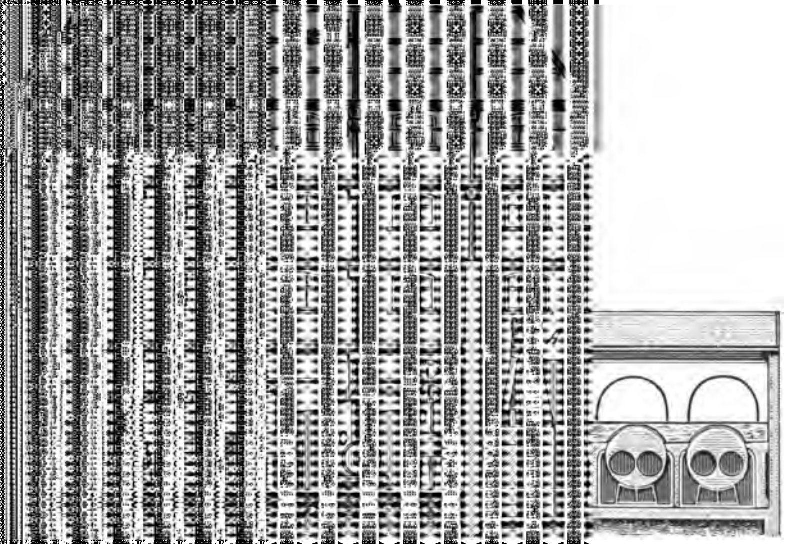
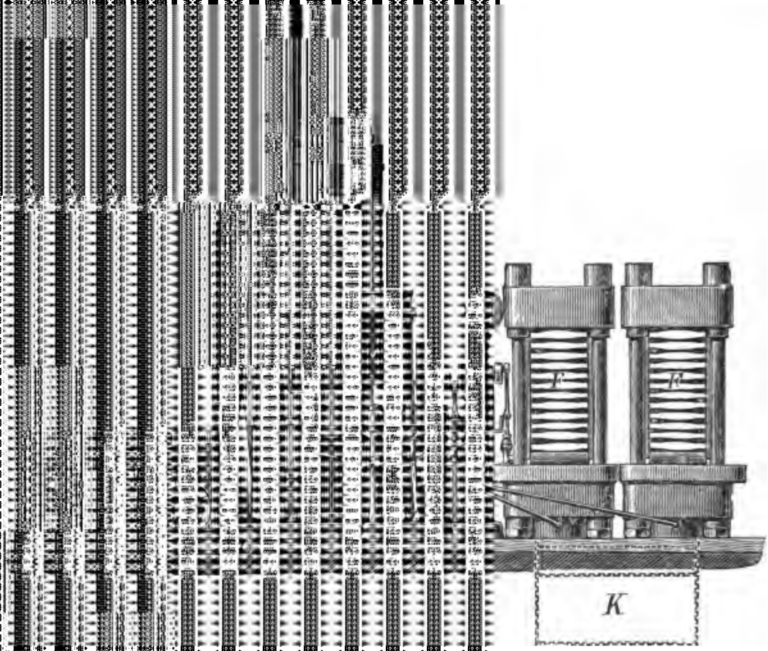


nstruction.

arranged near working them. Mill" depicted lly as possible provided with ; they work best arranged is the simplest [illegible] designed; including [illegible] und space of 6 × 3·75 [illegible] is readily accessible; [illegible] stallation, completely [illegible] of the building, etc. [illegible] driven by cog-wheel [illegible] engine, on the same



[illegible] Oil Mill.

[illegible] stability in the whole [illegible] nt of the roller-mill, [illegible] ws and elevators) for [illegible] s and the heating pan [illegible] diminished in number, [illegible] minimum. Also, the [illegible] ing parts are reduced.

A wooden hopper is usually placed over the rollers, but if an elevator is required to feed the seed to the rollers, it can be provided at a very small cost. The rational methods for heating and moistening the seed meal in the heating pan deserve special attention; for these purposes an improved steam jacket and a steam spray are used. The patented grinding machine for the cake trimmings, which is worked in conjunction with the trimming machine, is very efficient. The cuttings are rapidly ground to a sufficiently fine meal, which can at once be conveyed to the heater. Thus the heavy and expensive edge-runner mills hitherto usual are dispensed with, and there is considerable saving in power. The capacity of the installation for linseed, which in this case is only once pressed, is 7000 to 8000 kilos. per hour, according as the cakes are made thick or thin. The capacity for seeds which are twice pressed, is diminished by one-third to a half. Including a stoker, only three men are required in each shift to work the machinery of this mill, whilst with ordinary systems a dozen or more are required per shift. In the illustration

A denotes the vertical boiler, steam engine, feed heater, and feed pump.

B is the roller mill with five chilled iron rollers, 300 mm. in diameter, and 380 mm. long, with well-turned steel axles, wheel gearing, and feeder.

C is the wrought-iron steam-jacketed meal heater, with an insulating layer enclosed in sheet iron, improved steam sprayer for rationally moistening the seed in the pan, stirrer, pressure gauge, steam pipe, and condensed water outlet.

E is an improved steam cake-moulding and pressing machine, with an automatic cut-off for the hopper, which is placed below the heating pan, and two steel cake plates.

F F are two hydraulic presses, each producing fourteen cakes 700 mm. long, 280 mm. wide, and about 5½ to 6 kilos.

in weight, with a steel cylinder and piston 300 mm. in diameter, four steel columns and solid wrought grooved press plates.

G is an improved cake trimming machine with driving gear, six steel knives, conveyer, and steel shovels.

H is a patented grinding machine with chilled cast-iron rollers, specially grooved for the cake trimmings, with driving gear and feed hopper.

J is a set of hydraulic pumps with cast-steel cylinders, rectangular crank-axles of Siemens steel, connecting pipes, safety valve, release gear, and driving gear.

K is wrought-iron oil tank.

L is the massive cast-iron bed-plate with iron framework, gun-metal bearings, etc.

A process for producing oil, due to Spir ller, must not be omitted. The oleaginous seeds, kernels, etc., are ground and then treated with a boiling solution of 2 to 5 parts of common salt in 100 parts of water. Sugar, syrup, and similar substances may be substituted; they are, however, dearer, and are less valuable as constituents of the residue, which is designed for cattle food. The treatment of the oleaginous seeds with the salt solution may take place during the crushing under the edge-runner; a better method is to boil the seed with a sufficient quantity of salt solution and then bring it into press bags. A milky salt solution, containing almost all the soluble matter of the seeds, flows out under the slightest pressure; it is followed, under a greater pressure, by oil free from impurities, which floats above the salt solution; the latter is afterwards drawn off. The valuable vegetable matter so obtained is filtered off or allowed to deposit, and then added to the press residues. The salt solution may be used again. The press residues were formerly treated with hot water; this has been abandoned, since it makes them liable to decomposition. The residues obtained in the new process keep better in consequence of the salt, are better liked by cattle, and more easily digested than the residues obtained without salt, and especially than residues extracted by carbon bisulphide. For this process no heavy presses or extractors are required; it is sufficient to crush the seeds and press in a simple inexpensive press, when a pure oil is obtained, which is already free from mucilage and albumin. The residues are converted into a valuable food by the rupture of the plant cells and the swelling of the woody fibre, especially if the vegetable matter carried off by the salt solution is added. A proper amount of oil is left in the cakes after once pressing without hydraulic pressure. In the case of edible oil, there is the further advantage that the salt solution removes from the seed husk, together with the above-mentioned vegetable matter, the coloured impurities of bad taste which are transferred to the oil and injure its flavour when the seed is simply warmed. Thus by once pressing a pure and fine oil is obtained.

The process of Graham for obtaining oil consists in lightly roasting the fruits in a certain manner at 150° to 180° C. The kernels, after they have been separated from the husk, are then ground, the meal brought into a mixing vessel, and cold or hot water poured over. Whilst the water is being slowly added, the oil begins to separate from the thick mass, and rises to the surface in consequence of its low specific gravity. About 1 kilo. of water is required to obtain 1 kilo. of oil. The yield of oil varies somewhat according to the temperature of the mixture; the largest quantity is obtained at about 55° C., whilst at 4° C. 3 to 4 per cent. less is extracted. By this process not the whole of the oil contained in the seeds or nuts is obtained; the best result, which was attained with nuts containing 45 per cent. of oil, was about 33 per cent. The residual oil is not, however, lost; the most varied products can be obtained from the residues.

Oil is obtained by this new process more quickly and cheaply than by any other hitherto known. The oil is obtained completely clear and pure, so that no further arrangements are required for refining and filtering. Foreign constituents can be very readily removed simply by settling.

Oils may be partially or completely produced from seeds or fruits by the extraction process, according as the seed meal is extracted without previous pressing or the pressed cakes, after yielding the first pressure oil, are crushed, ground to a meal, and then completely extracted. The oils are extracted by readily volatile substances, which must possess the property, whilst they dissolve the fatty portions of the meal, of leaving undissolved other constituents, gums, colouring matters, etc. The solvents must also be readily and completely removed from the oils by heating and distillation, so that they are entirely regained, and do not leave the smallest amount in the oil. The solvent must be of such a nature that, after distillation from the oil solution, it has imparted no odour or flavour to the oil, which is obtained completely pure. The solvents used are carbon bisulphide, canadol, benzene, petroleum ether (benzine) and ether; recently carbon tetrachloride and sulphur dioxide have also been proposed. Carbon

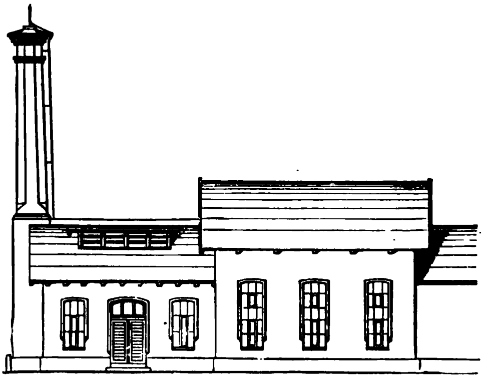


FIG. 9.—Oil-Extracting Works. Façade.

tetrachloride has none of the disadvantages of carbon bisulphide—unpleasant and harmful smell, inflammability, and the property of imparting an unpleasant flavour to the oil; it has a pleasant smell, is not poisonous, at least not in an ordinary diluted state, its vapours are not inflammable at ordinary temperatures, its boiling point is low, and yet so high that at the ordinary temperature loss through evaporation does not occur. It is not liable to decompose, leaves in the extracted oil no unpleasant smell or taste, and possesses great solvent powers. The oil extracted by means of carbon tetrachloride has a better colour than that extracted by carbon bisulphide, probably because the latter forms sulphur compounds which are generally dark in colour. The apparatus and process used with carbon tetrachloride are the same as with other solvents.

The process with sulphur dioxide depends on the hitherto unknown fact that compressed liquid sulphurous acid (SO_2) is a good solvent for oils and fats. It does not mix with oils and fats in every proportion to a homogeneous liquid, but the solubilities are such that the extraction of oleaginous materials by means of liquid sulphur dioxide can be effected in the same manner as with petroleum spirit and carbon bisulphide, and in a shorter time. So long as the oil is in excess, reckoning by volume, the mixture is homogeneous; in spite of this apparent drawback there are such advantages that the disadvantages are outweighed. In the first place, the danger of fire disappears; in the second, the sulphur dioxide, in consequence of its low boiling point ($-10°$ C.), can be much more easily removed both from the oil and the extracted material, and without the use of steam, which is a particular advantage when the extracted substances would be injured by the application of heat or by moistening with steam. The total separation of the sulphur dioxide from the oil is far more easily accomplished than with

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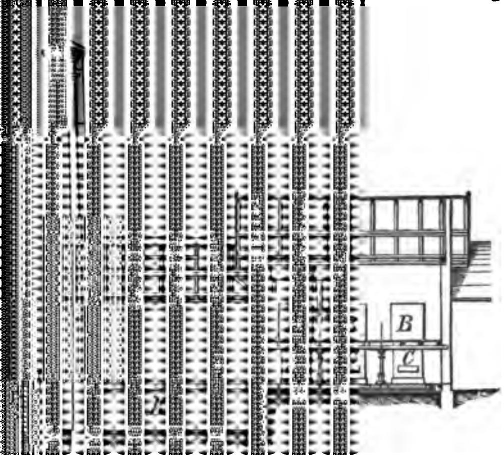

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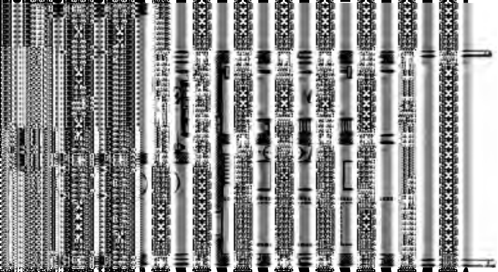

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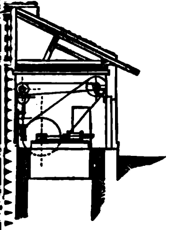

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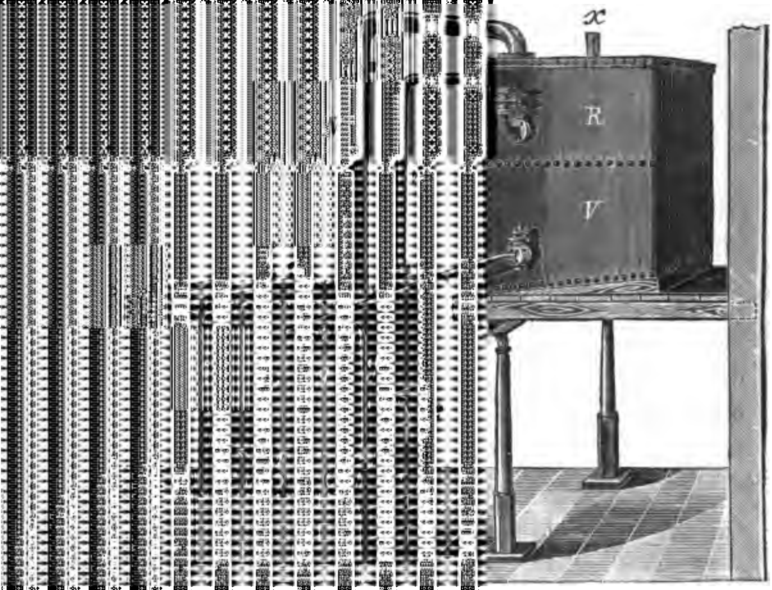


[illegible] lner and Merz.

[illegible] 2 × 20 metres; in it, [illegible] of 6 cubic metres [illegible] or 25 tons of cake [illegible]

[illegible] cted in Fig. 13. The [illegible] steam coil, contains [illegible] rough the manhole *d* [illegible] from the reservoir V, connected with the condenser R, solvent is allowed to flow into L; the solution of oil is then drawn off by the syphon *ggg* into M, as soon as its level rises above *g*. In M the solvent evaporates, the vapours rise round the walls of L, the contents of which they warm; they then reach the return condenser N, where they are condensed. The warm liquid flows back into L, whence it is again carried over into M as soon as the level reaches *g*. This automatic process is not interrupted until a sample taken at *h* indicates that the extraction is finished. The cooling water in N is next cut off, the vapours then rise from L to N, thence pass to the condenser R, and collect as liquid in the reservoir V. The final portions of the solvent are driven both out of the extract and the extracted material by direct steam. The oil is run off at *u*, and the extractor L emptied through *e*. The apparatus may also be used for continuous extraction as well as for the intermittent process just described. In that case the outflow of the oil solution is so regulated that the evaporated and recovered solvent maintains a constant level in L.

The working expenses may be calculated as follows:—

		s.	*d.*
Steam: 4½ tons of coal at 16s. per ton . .		72	0
Benzine: 4 cwt. (loss) at 10s. per cwt. . .		40	0
Labour: 1 fireman, 1 extractor, 3 labourers .		28	3
Various expenses and depreciation . . .		11	9
Working expenses per 24 hours .	£7	12	0

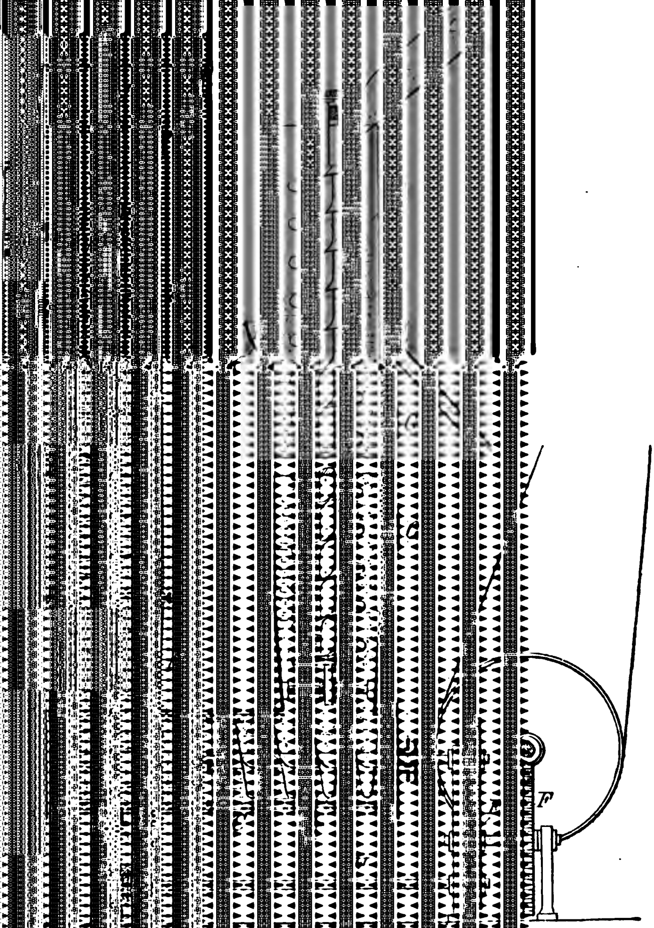
This is equal to 5¼d. to 6¼d. per cwt. of seed, or 3¾d. per cwt. of cake.

A good process of extraction by benzine (petroleum spirit) is unquestionably to be given the preference over the best system of pressing, for the yield is not only considerably larger as compared with the best pressing, but the extracted oil is more pure than expressed oil. The mucilage and albumins, which escape in pressing, and especially in hot pressing, are not taken up by the solvent. These substances remain in the extracted residue and increase its nutritive value. The residue, which has no trace of smell or taste of the solvent, is readily eaten by cattle; the meal has an odour and taste resembling new bread. Another important advantage is that this meal can be stored for a long time without deterioration, whilst press cakes are soon spoiled by the oil in them gradually turning rancid, when the cakes become uneatable. The change is the more rapid the more oil they contain.

Of great importance is the process which combines pressing with extraction, in which the seed is pressed cold, the cakes ground and then extracted.

Extraction apparatus of Prosper Monnet.—This apparatus consists essentially of two upright cylinders, connected together after the manner of pipes. The motion of the liquid is caused by a difference in level, whilst the solid material under treatment is moved by screws, which revolve at a speed depending on the rate of outflow of the liquid. The continuous extraction is carried out in such a manner that the smallest quantity of solvent produces the most concentrated extract possible. The cylindrical arrangement of the apparatus also enables the solvent to act upon the material under a pressure which varies according to the height of the cylinders and the nature of the material. This circumstance accelerates the extraction of the oil.

The apparatus is shown in Fig. 14 in vertical section. The vertical cylinders A and B, of height equal to ten times the diameter, are somewhat narrower towards the bottom, they stand upon a horizontal cylinder C, which is equal in diameter to the vertical cylinders at the junction. The material to be extracted enters the cylinder A through the hopper H, [illegible] sary, by the [illegible] carries the [illegible] B, in which



[illegible] net.

[illegible] falls out into [illegible]

[illegible] part of the [illegible] tes in a rose.

The liquid moves in the opposite direction to the material under extraction, and rises in A to flow out into the channel K through holes NN, placed at a lower level than that at which the liquid enters B. The screws D and E are actuated from the outside by an ordinary means of transmitting power, as by the wheels FF shown in the drawing. The screw M may be omitted in all cases in which the material to be extracted does not cake together. If it is necessary to use it, it may be moved from above, mechanically or by hand. In any case the horizontal screw D must be able to move the material forward more rapidly than M, and E than D, so that stoppages do not occur.

The process of the extraction is then as follows:—The ground oil seed comes through the shoot H into A, and the solvent, carbon bisulphide, carbon tetrachloride, or benzine, into B. Steam is admitted into the jackets (not shown in the figure) in such quantity as is required. The screws receive such a velocity that the meal remains in the apparatus a sufficient length of time to be completely extracted. Thus the meal admitted into A must, on its exit from B, be quite free from all soluble matter, which it has given up in its passage through the apparatus, whilst the solvent should run off by L, saturated with oil, and be lead to a distilling apparatus.

Oil Cakes and Meal

The residues from the production of oil are oil cakes from pressing and oil meal from extracting. Both are valuable as cattle foods, since, in addition to the nutritive matter they contain, they also possess a pronounced dietetic action, which they owe principally to the mucilage they contain. The two products differ, in the first place, in form, as expressed by the terms cake or meal; the other chief difference is in their content of oil, which is for the former

7 to 20 per cent., for the latter 2 to 4 per cent. All meals and cakes are used as fodder for cattle, and in consequence of their high percentage of proteïns, fat, and phosphoric acid, produce a better yield from the cows, and also a better manure. Their food value is not less than that of the seeds, since the nutritive constituents of both are digestible in the same manner (78 to 98 per cent.). In the case of milch cows, oil cakes generally act favourably upon the yield of milk, both as regards quantity and quality (percentage of fat).

The cakes and meals which have to be considered in connection with the drying oils are (1) linseed cake and meal, (2) hempseed cake, (3) poppy cake, (4) sunflowerseed cake, (5) candle-nut cake—

(1) **Linseed cake and meal.**—Linseed cake is an excellent food for calves and breeding cows; it is too dear for ordinary use. It contains—

10–13	per cent. of	water.
30–35	„	proteïns.
9–13	„	oil.
26–30	„	extractive matters free from nitrogen.
7–10	„	woody fibre.
6–9	„	ash.

489 analyses of linseed cake gave an average as follows:—

11·82	per cent. of	water.
28·70	„	proteïns.
10·74	„	oil.
9·35	„	woody fibre.
7·37	„	ash.
42·13	„	carbohydrates.

On the average, 86 per cent. of the proteïns, 90 per cent. of the oil, and 80 per cent. of the extractive matter free from nitrogen were digestible.

Linseed meal extracted by carbon bisulphide is not so readily eaten by cattle as linseed cake; it contains only 2 to

3 per cent. of oil. There is sold, under the name of linseed meal, a meal which has the same composition as linseed cake. Linseed cakes and meal are liable to various adulterations; the adulterants are the most varied fodder refuse of small value, for example, damaged flour or meal, rice meal, beech-nut cake, unshelled earth-nut cake, etc. In examining linseed cake or meal, the natural impurities of linseed must be remembered, it contains always 3 to 8 per cent., and occasionally 50 per cent. of foreign seeds, principally seeds of the cruciferæ, dotter, cereals, etc.

(2) **Hempseed cake.**—This cake, the residue of hemp seed, which contains 30 to 36 per cent. of oil, is principally produced in the Baltic provinces of Russia, more rarely in Germany; only in the former district is the oil produced in considerable quantity. The cakes exported from Russia are used almost exclusively in East and West Prussia; they contain—

8·0–12·0	per cent. of	water.
25·0–33·5	„	proteïns.
4·3–10	„	oil.
16·0–26·0	„	extractive matters free from nitrogen.
23·0–26·0	„	woody fibre.
5·0– 7·0	„	ash.

Hempseed cakes are less valuable than other oil cakes in consequence of their high percentage of woody fibre, also in warm weather they readily deteriorate and turn mouldy. In Eastern Prussia they are used almost exclusively as food for horses and sheep.

(3) **Poppyseed cake.**—The residues from poppyseed, which contains from 38 to 44 per cent. of oil, have hitherto found a restricted use in Germany as fodder; they are more largely used in South and Middle Germany than in North Germany. The percentage composition is—

10·0–15·0	per cent. of	water.
28·5–33·5	„	proteïns.
6·6–14·0	„	oil.
23·0–29·0	„	extractive matters free from nitrogen.
10·0–13·0	„	woody fibre.
6·0– 7·3	„	ash.

The digestibility of poppyseed cake has not been investigated, but it may be assumed that the nutritive materials will be digested in a similar manner to the corresponding constituents of other oil cakes. Poppyseed cakes are said to make the animals sleepy and stupid, they are therefore more suitable for fattening than for milch cows; the milk readily acquires an unpleasant taste.

(4) **Sunflowerseed cake.**—These cakes are also valuable as cattle food; they contain—

10·00	per cent. of	water.
36·55	„	crude proteïns.
10·50	„	crude fat.
23·97	„	extractive matters free from nitrogen.
9·25	„	woody fibre.
9·73	„	ash.

90 per cent. of the proteïns, 88 per cent. of the fat, and 71 per cent. of the nitrogen-free extractive matters are digestible.

(5) **Candle-nut cake.**—These are an extremely concentrated food, they should be a valuable addition to rations poor in proteïns; they contain—

7·22	per cent. of	water.
54·38	„	crude proteïns.
9·15	„	crude fat.
15·61	„	extractive matters free from nitrogen.
4·58	„	crude fibre.
9·06	„	ash.

(6) **Walnut cake.**—This contains—

13·7	per cent. of	water.
34·6	„	crude proteïns (31·1 per cent. digestible).

12·5	per cent. of	crude fat (11·2 per cent. digestible).
27·8	,,	extractive matters free from nitrogen (28·2 per cent. digestible).
6·4	,,	crude fibre.
5·0	,,	ash.

(7) **Pumpkinseed cake** contains—

12·0	per cent. of	water.
55·6	,,	crude proteïns (50·0 per cent. digestible).
11·4	,,	crude fat (10·3 per cent. digestible).
8·0	,,	extractive matters free from nitrogen (9·7 per cent. digestible).
4·9	,,	crude fibre.
8·1	,,	ash.

THE REFINING AND BLEACHING OF THE DRYING OILS

It is easily comprehensible that the drying oils, as they are produced, whether obtained by expression or extraction, must contain a quantity of mechanically admixed impurities. The amount is greater in the former case than the latter, since in the extraction process only substances soluble in the solvent are withdrawn, but in pressing, in consequence of the great pressure employed, other substances accompany the oil—colouring matters, gum, mucilage, and albumins. Freshly pressed oil is always turbid; when left at rest for some time it deposits many of the mechanically admixed impurities. The oil then becomes clear, but still obstinately retains impurities, which become evident on heating, when they separate. The simplest method of purification is to leave the oil in a large tank to deposit the grosser impurities, then to filter (through a suitable filter, of which there are many different constructions), and to bring the oil into the market in the resulting bright transparent condition. Nothing more can be demanded of commercial linseed oil than that it shall be pure and clear; it is then sufficient for ordinary use in paints and for boiling. The case is somewhat different with "tanked oil" and "varnish oil" for the manufacture of oil varnishes, which is not only required to be quite bright and free from foot, but also not to throw out a foot when heated to 270° C.

The substances which accompany expressed oil are generally designated "mucilage." The nature of this mucilage has not yet been decided. Weger makes the following remarks concerning linseed oil mucilage:—In the manufacture of oil varnishes, "varnish linseed oil" is used; by this term is understood an oil which, when rapidly heated to 280° to 320° C., does not "break," *i.e.* does not separate any solid substances. If fresh raw linseed oil be tested by rapidly heating it in a test-tube, a turbidity generally appears at 250° C., and on further heating large flocks separate, which fill a considerable part of the tube. Ordinary commercial linseed oils (with the exception of "varnish oil," which has undergone some chemical or mechanical treatment) almost always show this coagulation on rapid heating. It should be observed that much depends on the rapidity of the heating; if the temperature rises slowly the separation does not always occur. The nature of the separated substance, whether mucilage, albumin, or something else, is as little known to-day as in Mulder's time; it is generally known as mucilage. It is often present in absolutely bright tanked oils.

Mulder admits the possibility of the presence of mucilage and albumin in cold-pressed turbid oils alone; this is incorrect without doubt. If Mulder could find by his own method no mucilage or albumin in commercial linseed oil, he must accidentally have used an exceptionally good oil, or his methods for detecting these substances must have been unreliable. The quantity of mucilage is certainly small, for although in the test the tube often appears to be almost filled, very little is obtained on filtering. Mucilage is also present in other oils, *e.g.* rape oil, and separates on rapid heating.

It was and is yet often maintained that the mucilage affects the drying of linseed oil and makes it unsuitable for the manufacture of varnish. So far as the drying is concerned this is quite untrue. Already Mulder had said that linseed oil filtered through charcoal — which must extract even dissolved mucilage, since oil filtered through charcoal does not "break" on heating—dries as well or as badly as before. The contrary has since been repeatedly maintained, and Mulder's views have also been upheld. Weger has never been able to observe any difference between the drying of a clear mucilaginous linseed oil and of the same oil freed from mucilage; the latter dries just as often a little better as a little worse. On the other hand, Weger heated a large quantity of oil, collected the mucilage on a filter, and freed it from oil as far as possible, but very incompletely, on a porous plate. The residual substance was a gelatinous transparent mass of yellowish colour, which became covered by a skin in a few days. When spread upon glass the mucilage dried not only as rapidly, but on the average a day sooner than the oil now free from mucilage. This might perhaps be due to a partial oxidation of the substance by long standing upon the filter; however, the material did not appear sticky and thick when rubbed upon glass, but behaved like linseed oil. However that may be, it is decided that mucilage and mucilaginous linseed oil do not dry more slowly than oil free from mucilage. From the observations made during the experiments, it appears that the absorption of oxygen by the mucilage is smaller than by the oil. It should be observed that the refraction of such mucilage, still containing oil, as observed in Zeiss's butyrorefractometer, is 77°·5, whilst the original oil showed 72°·4, both measured at 40° C. The mucilage is not removed by long standing, or at least not always, when no special treatment is associated with the storage.

One of the most important impurities in linseed oil is the fatty oil of mustard seed. This oil is almost always contained in linseed oil, in good qualities, however, in very inconsiderable amount. The seeds of the mustard and allied plants are already contained in the linseed used by the crushers, the expressed oil then naturally contains mustard oil. If the percentage of this oil is small it interferes little with its suitability for boiling; but if a large quantity be present the boiled oil loses its property of drying rapidly. A large proportion of mustard oil in linseed oil makes itself perceptible by the deposit of a yellowish foot when the oil is kept for several days in a moderately warm room (10° to 12° C.). The percentage of fatty mustard oil may be more accurately estimated by means of the elaïdin test; after 2 to 3 days elaïdin separates. Pure linseed oil does not solidify even after a longer time.

In the manufacture of varnishes linseed oil free from mucilage, which does not "break" on heating, is used. It is obtained by a process of refining, or by tanking for a sufficiently long time. For tanking large open tanks are employed, at a moderate temperature of at least 15° C.; if it is in any way possible, sufficient quantities of air should be conveyed to the oil by fans or blowing machines. This process is very slow and is often replaced by chemical or mechanical refining processes, followed by filtering.

The essential of all refining processes is to mix the oil as intimately as possible with the refining agent—acids, salts, etc.—so that all portions of the oil may come in continued contact with it. The same is the case in bleaching, where an intimate emulsion must be formed. It would overstep the limits of this book to describe in detail the mechanical mixing arrangements used. It must suffice here to say that they are very varied and may be classified as follows:—

(1) Stirrers, in which the stirring arm is attached to a

horizontal or vertical axis, moved by power, and thus accomplishing the mixture of the oil with solids or liquids.

(2) Apparatus, in which air is blown into the oil or drawn through it, by either of which means an intimate mixture is produced. The process is much more efficient than with mechanical stirrers, and the air also exerts a bleaching action, due to oxidation, which can only be an advantage in the case of a drying oil intended for painting purposes.

(3) Centrifugal emulsifiers, which have found little employment up to the present, but are capable of replacing all other mixing apparatus. The advantages of centrifugal emulsifiers are uninterrupted working, great capacity with small expenditure of power, the small space required, saving in refining materials, better quality of oil, and the possibility of using freshly expressed oils.

The various processes of refining are accomplished by the use of dilute acids, salt solutions, substances in the form of powder, such as magnesia, other metallic compounds, and earths. The drawbacks of the processes in which aqueous liquids are used, are principally the difficulty of separating (as a rule) the two liquids, and the loss which occurs owing to the formation of emulsions. When the separation of the two layers is complete after rather long standing, there remains between the oil and the liquid used for refining an emulsified layer which separates with difficulty, so that loss of oil occasionally makes the process somewhat costly. Alkaline liquids are to be avoided in refining, they saponify the oils and make necessary an addition of acid to bring about separation. Refining with acids, zinc chloride, etc., effects a decomposition or carbonisation of the organic matters suspended in the oil; refining by means of solids, such as magnesia, fuller's earth, and powdered mineral substances, produces only a mechanical purification; these heavy bodies in settling to the bottom drag with them the particles suspended in the oil.

All oils which are refined by acids or chemicals in aqueous solution, require careful washing with water until they are quite neutral, otherwise difficulties will occur in the subsequent use of the oils.

Refining with sulphuric acid.—According to Wasl, 1 kilo. of oil of vitriol is used to 400 to 500 kilos. of linseed oil; it is added in a very thin stream under constant stirring. After long mixing, boiling water, equal to one-third of the weight of the oil, is added, the whole well mixed and allowed to settle. After completely separating the acid liquid from the still turbid oil, the latter is drawn off into another vessel, and 3 per cent. of dry powdered common salt is added. The salt withdraws the water still contained in the oil and settles to the bottom on account of its greater specific gravity; the oil is then filtered through bags containing wheat bran. When these filters have been used they are pressed, the oily bran used as fodder, and the bags cleaned by boiling with alkali.

In another process, after the oil has been thoroughly treated with sulphuric acid, it is not immediately mixed with water, but is allowed to stand 12 to 15 hours. After this time the oil can be drawn off clear and pure, whilst the slime remains thick and fast at the bottom. The oil is then treated with a boiling 5 per cent. solution of common salt for several hours until a slight white foam appears on the surface, when the oil is drawn off and filtered.

Wilke uses 3 parts (by weight) of sulphuric acid to 400 parts of oil, which is treated with the acid for 5 hours; 3 parts of clay, 7 parts of quicklime, and finally 1000 parts of water are added and mixed with the oil for 3 hours at 100° C. In Logan's process 5 parts of strong sulphuric acid, diluted with 5 parts of water, are used to

400 parts of oil; the acid is added in small quantities and the mixture kept in constant motion for several hours. It is then allowed to settle and steam led in, finally the oil is left at rest to brighten.

In place of sulphuric acid, zinc chloride solution may be used in the proportion of 1 to 15; it exerts the same action on the oil without possessing the disadvantages of the acid.

The refining of linseed oil by means of ozone.—According to Engleduc, linseed oil can be advantageously refined by means of ozone. The oil is freed from moisture and mucilage, which would otherwise be accomplished, at the cost of the colour of the oil, by heating or by very long standing. In the ozone process the valuable properties of linseed oil are entirely retained, whilst the oil gains but little in viscosity. The process is complete in 2 to 3 hours. Besides the apparatus which produces the ozone, there is required only a shallow steam pan to contain the oil; it is provided with a tinned perforated coil through which the ozonised air is introduced, it then rises in bubbles through the oil. In the preparation of "boiled" oil various driers are dissolved in the raw oil and the mixture treated with ozone in a separate apparatus at a certain temperature for 3 hours. The oil obtained possesses in a high degree the properties of an ideal drying oil—rapid drying, consistence, hardness, elasticity, and pale colour. The temperature required is low, and thus no loss occurs.

According to another account, linseed oil is heated to the required temperature in a steam pan, into which open pipes for the introduction of ozonised air. The oil loses its water and all other impurities, retains its properties, and thickens readily. The air to be ozonised is pumped through a drying chamber, in which are shelves covered with quicklime. It is then conducted through the ozoniser and compressed into a strong iron reservoir, from which it is divided amongst the various refining apparatus. In order to refine 1 ton of linseed oil, 310 to 315 cubic metres of ozonised air are required on the average. According to Lewkowitsch, this volume of air can be ozonised by means of Yarnold's ozoniser at a cost of about 1s. 8d. In small works, which treat about 5 tons of oil per day, the cost of refining would not exceed 7s. 6d. per ton. The advantages of the process are decrease of waste, rapid refining, production of a faultless product, and exclusion of danger of fire.

The refining of linseed oil by means of calcium carbide.—According to the patented process of de la Roche an ordinary mixing apparatus is used; when this has been filled with the oil, 10 per cent. of powdered calcium carbide is added and the stirrer kept in motion for 1 hour. Amid an evolution of acetylene the oil is converted into a greenish mixture in consequence of the changes caused by the free lime produced. As soon as the exhaustion of the moisture causes a cessation of the evolution of acetylene, the oil is allowed to settle and decanted or filtered through filter-presses from the deposit. The oil obtained is almost colourless, clear, bright, and entirely free from water and acid. It is boiled in the ordinary manner, with an addition of 3 to 5 per cent. of lead or manganese compounds. The cakes taken from the filter-presses still contain undecomposed carbide; they can be used to produce acetylene to light the works. The water added for this purpose contains some caustic alkali, which saponifies the oil enclosing the particles of carbide. By this method of utilising the press cakes the cost of the process of refining is diminished.

Wet process for clearing linseed oil.—Direct steam is passed for 1 to 2 hours through a mixture of equal parts of raw linseed oil and hot water. The oil is then allowed to stand until separation occurs. The water collects at the bottom, the clear oil floats above, whilst the precipitate forms a white layer between the two liquids. After standing 5 days the clear upper layer of oil is drawn off, brought into a pan, and heated for 2 hours at 220° F., to drive off water. Many manufacturers add ·1 per cent. of sulphuric acid to the water used in this process, the oil is then bleached and clears somewhat more rapidly. Also, by the addition of a few pounds of Fuller's earth or lead sulphate, the colouring matter, as it settles, takes with it the albumins from the oil, and thus accelerates the process. The foots can be sold to putty makers, who buy them on account of their low price. The clear oil produced by this wet process is distinguished by a very pale colour; it approximates to oil purified by magnesia. In case the cost of the process is not the first consideration, especially fine oil can be obtained by first bleaching the oil in the presence of acid by the wet process, and then refining by means of magnesia.

Refining by means of cellulose. — If linseed oil is allowed to stand undisturbed in a glass cylinder from 2 to 3 months, water deposits first, then a somewhat thicker layer separates, voluminous, flocculent, and heavy; this is the substance called "mucilage." At the ordinary temperature about a week is required for the deposition of the water, and 3 months for the mucilage; in a year all the insoluble constituents have settled, inclusive of coagulum and globulins; pure linseed oil remains above the deposit. The specific gravity of the globulins is, however, so near to that of linseed oil that the least rise in temperature causes their partial or complete re-solution. Endeavours to filter linseed oil through thick paper are without result, since the mucilage, globulins, and coagulum are soluble in the oil, and simply filter through. If 60 kilos. of freshly expressed linseed oil, as it comes from the press, be mixed with about 0·5 kilo. of neutralised cellulose, and a test portion withdrawn after stirring for about 15 minutes, it will be seen that the cellulose is gradually absorbed by the oil, and the mixture, instead of being turbid, is transparent and clear. It appears as if the cellulose were dissolved by the oil; the molecules of the cellulose are, however, only mechanically mixed with the oil, forming a transparent varnish-like mass. If the mixture be allowed to stand some moments the cellulose is reprecipitated, and, what is most important for our purpose, brings down with it the mucilage, globulins, coagulum, and water. This process requires about 12 hours. In order to work profitably a filter-press must be used to separate the oil from the cellulose. In this apparatus a gradual separation takes place, the cellulose and the impurities of the oil forming a cake, whilst the purified oil leaves the press clear and bright. For this purpose the cellulose must be absolutely neutral and free from resins and fats. It must have been prepared by the most careful washing and purification; if it contains only a trace of resin or fat it will dissolve in the oil and make it still more turbid.

Nördlinger's refining process.—Nördlinger avoids the disadvantages of ordinary refining processes and the use of mechanical methods by treating the oils with solutions in oils of certain reagents—in particular, the solutions of certain salts of the fatty and resin acids, benzoic acid and its homologues. So far as it is at present known, the solubility of the salts of the fatty acids in vegetable oils increases with the increasing molecular weight of the fatty acids. From butyric acid upwards the salts generally dissolve in 10 to 20 parts of vegetable oil at temperatures of 100° to 200° C. In regard to the solubility, the composition of the vegetable oil is of little importance; the salts in question dissolve to an equal extent in linseed, sesamé, poppyseed, rapeseed, cedarnut, cottonseed, etc., oils.

The 5 to 10 per cent. solutions of the alkaline salts of the fatty acids containing more than 4 carbon atoms, are at the ordinary temperature more or less thick viscous liquids, which, however, do not form any solid deposit (the corresponding salts of oleïc, abietic, and sylvic acids and their homologues behave in the same manner). The solutions of the metallic salts of these acids in oils are, at the ordinary temperature, similar in consistence to lard, or a portion of the salt dissolved at the higher temperature has again crystallised out. Solutions of 5 to 10 per cent. strength of the zinc, cadmium, iron, copper, manganese, and lead salts of each of these acids are clear at 40° C., or at most at 100° C., the nature of the vegetable oil used as solvent being unimportant. The lime, magnesia, baryta, and alumina salts also dissolve in vegetable oils, but do not possess in the same degree the peculiar property of the salts of the heavy metals of precipitating the mucilage.

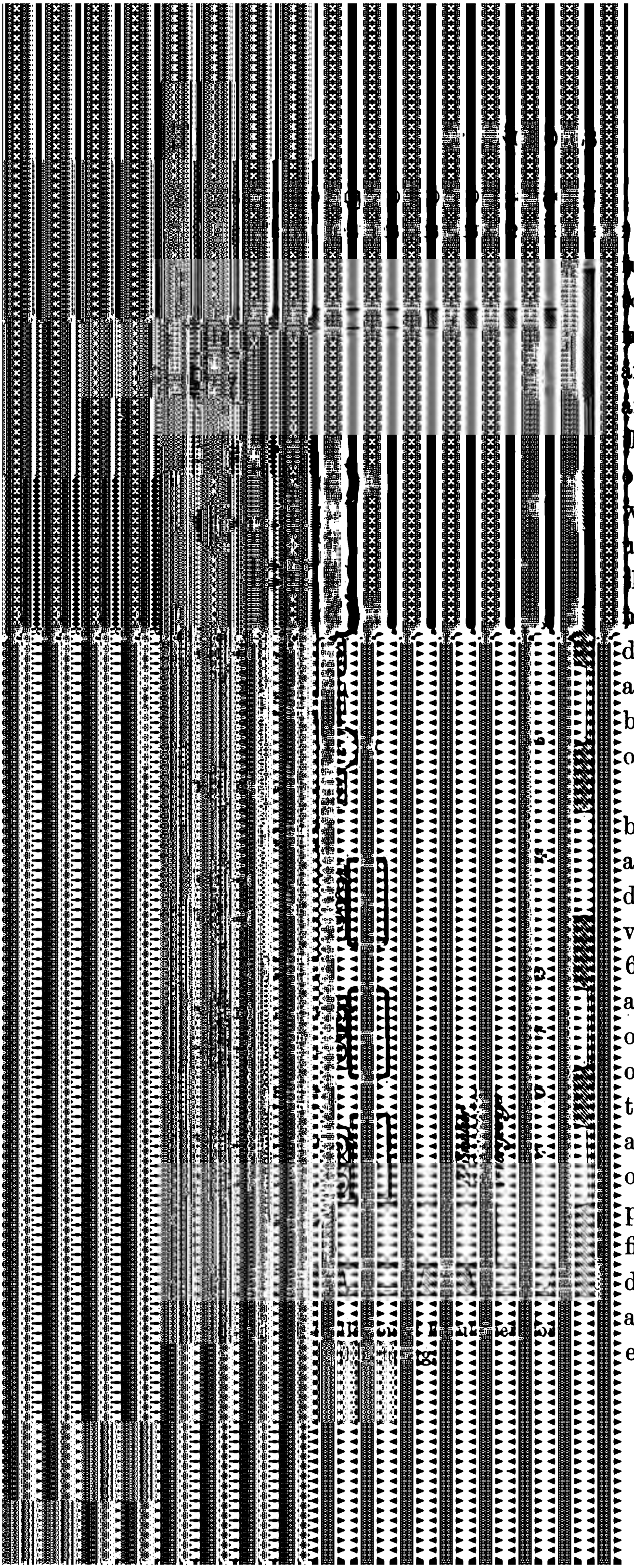
If small quantities, 5 to 10 per cent., of the solutions in oils of the heavy metal salts of the acids mentioned be added to ordinary vegetable oils a clear solution is at first formed; after some time the mixture becomes turbid, especially if air has access, and the mucilage is gradually precipitated in combination with the previously dissolved metallic salts in fine flocks, more or less coloured. According to Nördlinger's view, a chemical decomposition does not take place, but the particles of mucilage are apparently weighted down by the metallic compound and caused to separate, as is also the case with certain colouring matters. The turbid oils are freed from the turbidity by filtration or settling whilst warm.

The process is now as follows:—The salts of the heavy metals (iron, manganese, lead, copper, zinc, etc.), with the higher fatty acids, the substituted fatty acids, benzoic acid, etc., are dissolved by heating to about 150° C. in about 10 to 20 times their weight of vegetable or rosin oil. The solution is slowly cooled and the clear liquid drawn off from the deposited insoluble portions while still warm (50° to 80° C.). Such solutions, called "refining oils," are as follows: 5 per cent. solution of zinc oleate in cottonseed oil, 10 per cent.

solutions of lead oleate or of iron stearate in sesamé oil, or of iron or manganese resinate in linseed oil.

The "refining oil," if not clear and fluid at the ordinary temperature, is liquefied by gently heating, and mixed with 10 to 20 or even 30 times its weight of the vegetable oil to be refined. A clear solution is formed, which, after standing some time, becomes turbid, especially if exposed to the air. The impurities deposit together with the metallic salts, and are then removed. Ferric manganese resinate is used for refining linseed oil.

Centrifugal emulsifiers for refining.—The process for refining oil by means of the centrifugal emulsifier consists in passing it continuously through a system of emulsifiers and separators, and in this manner subjecting it to the action of chemicals and to the necessary washings. The combination of one emulsifier and one separator forms a "washing element." In cases in which the emulsion separates so readily that no centrifugal separator is required, a Florentine receiver is substituted. The number of elements which are combined in a battery depends on the number of washings required. As a rule the emulsion flows directly from the emulsifier to the separator, since the reaction between the fluid or dissolved refining agent and the constituents of the oil to be removed takes place immediately, in consequence of the extremely intimate contact. If, in isolated cases, a longer action be desired, two tanks are introduced into the series to take the mixture in turn. For example, if the mixture is to stand 24 hours, the tanks must be made so large that each can take a day's product. Whilst the emulsion is allowed to run into one tank, the mixture from the other tank, made on the previous day, is treated in the separator. The proportions of the mixture are regulated either by suitably throttling the outflow openings of the two-chambered supply vessel by means of conical regulating valves, or, in large installations,



For rapid heating or introduced. Centriugal pumps are used o convey the liquid rom one element to nother, these pumps re driven direct from he shaft of emulsifier r separator, with vhich they work in nison. Eckenberg, he discoverer of this nethod, can effect any desired oxidation by air, or deodorisation by dry steam, by means of a gas emulsifier.

The capacity of a battery of emulsifiers and separators of ordinary dimensions varies from 3000 to 6000 kilos. per day, according to the nature of the oil, the manner of refining, the proportions of the mixture, and the combination of the apparatus. The plates of the emulsifier and the separator drum are made of acid-steel, which offers extraordinary resist-

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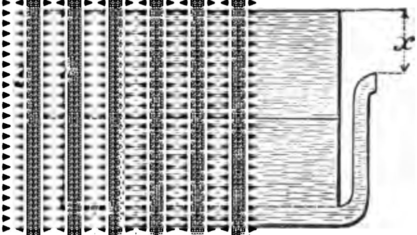

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of the vessel. If h represents the height of the flask from the edge to the entrance to the pipe (Fig. 16), x the distance from the orifice to the upper edge of the flask, y the height of the lighter liquid, s its specific gravity, and s_1 the specific gravity of the heavier liquid, then

$$sy + (h - y)s_1 = (h - x)s_1,$$

or,

$$y = \frac{x\ s_1}{s_1 - s}.$$

Thus, the specific gravities being known, y varies as x. The distance x can be so adjusted that the apparatus gives the greatest possible separation. When $y = h$, x is a maximum, therefore—

$$x\ (\text{max.}) = \frac{h(s_1 - s)}{s_1}.$$

If now x were made still larger, oil would escape with the heavier liquid by the side tube. If x were made too small, the emulsion would frequently not have time to separate, and a mixture of oil with the heavier liquid would flow over the edge. The distance y must accordingly be approximately calculated for each pair of liquids, oil and refining reagent, from their specific gravities, and afterwards so adjusted that the best effect is obtained in working. The position of the layer of contact in the separator, corresponding to the distance y, is independent of the proportions of the two liquids in the mixture.

But also when the apparatus has been properly adjusted it may happen that the emulsion does not properly separate. Especially when fatty oils are being washed by alkaline solutions, large quantities of oil frequently remain suspended in the liquid. There is a class of emulsions, which Eckenberg designates "critical," which not only pass through the separator unseparated, but, on the contrary, become still more stable. In such cases, by altering the concentration of the washing fluid or by working with warm liquids, more satisfactory results are generally obtained. A moderate addition of common salt or Glauber's salt is often of assistance, but the proportion of salt in the water should not be increased to such an extent that the soaps, which are being removed, are salted out. Thus a battery arranged for one oil cannot be used for another without adjustment of the conditions. In order to obtain the fatty acids and small quantities of oil contained in the wash waters, the latter are acidified, when the oil rises to the surface. The small quantities of oil which remain as emulsions in washing with water or dilute acids may also be recovered by one of the methods described by Eckenberg.

The emulsifier itself consists essentially of two flat plates of peculiar shape, placed opposite one another, with double rims ground perfectly plane, which slide one into the other. The lower plate is rigidly connected to the vertical shaft, which is prolonged beyond the upper surface of the plate, the prolongation having a conical boring narrowing above. The tube formed in this way conducts the fluids into the apparatus. For this purpose, immediately above the lower plate, with which it forms one piece, it is provided with two outflow openings, through which the liquids reach the space between the wide plates. On the exterior of this tube a screw thread is cut, upon which the upper plate is laid and then fastened down by two nuts. Three micrometer screws are placed in the rim of the upper plate, which enable the distance between the plates to be accurately and easily adjusted to any measurement. The axis is driven by means of a small turbine which is suitable for experiments on the small scale, but in working on the large scale it is driven by a rope. It runs at about 7000 revolutions per minute.

The plates of the emulsifier are surrounded by a fixed casing, the inner surface of which is covered with sheet lead. It catches the liquids mixed into an emulsion and conducts them away by means of a pipe to a tank or direct to a separator. Upon the cover of the casing is fixed a cylindrical vessel, open above, and divided into two chambers by a vertical partition. Into these divisions flow the liquids which are to be mixed, each division is provided with an outflow controlled by a valve, from which the liquids reach a common pipe opening into the upper open end of the emulsifier. By means of the valves the proportions of the two liquids are accurately regulated. To facilitate the adjustment, the spindles of both valves are provided with a scale divided from 0 to 100. In order to maintain a constant level, and therefore a constant outflow, both divisions are provided with floats.

When once set in proper working order the apparatus works continuously. Before the commencement, the time required for both liquids to run through the apparatus, which depends on the viscosity, must be found once for all by a preliminary experiment, which can readily be done by means of the cover of the emulsifier. This cover carries, as has been said, a vessel divided into two equal chambers by a vertical partition, each chamber having an outflow provided with a regulator. Both regulators must be arranged so that the two liquids pass through in exactly the proper proportions; this can readily be done by a few experiments. When the position has once been found the apparatus can be worked continuously, and need only be stopped when oils of considerably different viscosity are to be treated. In order that the emulsifier may be used conveniently in the oil industry, it must generally be combined with a separator, which is able in a brief time to part the otherwise stable emulsions; this is especially necessary when alkaline or neutral liquids are used.

The separator of de Laval may be used with advantage when oils rise so slowly out of emulsions that the ordinary arrangement, on the principle of the Florentine flask, consisting of a wooden tub with a separating pipe, cannot well be used. This separator is made in different sizes, with a capacity of 300 to 2000 litres per hour.

Extraction of the oil left in refining powders.—When oils are bleached and refined by substances in the form of powder, such as Fuller's earth, etc., the latter retain a considerable quantity of oil, up to 40 per cent. It is very difficult to re-obtain this oil, since the use of benzine, etc., is attended with danger of fire; the treatment with hot water is very tedious, and only partially effective. However, the oil may be completely separated by treating the refining powder under pressure at a high temperature, say 180° C. The oil then separates completely from the powder, which is at the same time purified and made again useable. A partial saponification in this process is of no practical importance, since the oil obtained can only be used by soap makers. By this means the oil is regained to fractions of a per cent.

THE BLEACHING OF LINSEED OIL

The requirements of the buyers (dealers and consumers) of all chemico-technical products continually become more exacting. The demand for a colourless or almost colourless linseed oil becomes greater and greater. The only substantial reason for the use of bleached oil is that paints ground in such an oil show a pure white colour, which pleases the eye of the buyer. However, the yellow or yellowish colour of linseed oil has no influence upon the pure white shade of a dried coating of zinc white, white lead, or permanent white, for with the drying of the paint the linseed oil (raw or boiled) bleaches so completely that not even the least shading of the white is to be observed. At the same time, there is also the advantage that a pure oil may be used, unaltered by the action of bleaching agents, which is generally not the case with paint oils bleached artificially and not by the simple action of light. However, buyers demand bleached oil, and consideration must be given to the demand.

The bleaching agents at our disposal are numerous, they do not all produce equally favourable results, and they also vary in their action upon the oil. In addition to light, which produces the most complete and finest bleaching of linseed oil, the following substances are used as bleaching agents:—

Chlorine or chloride of lime.
Potassium bichromate.
Potassium permanganate.
Hydrogen peroxide.
Sodium peroxide.
Sulphurous acid.
Sodium hyposulphite.

Other bleaching agents have also been proposed, which, however, do not give a good result.

Bleaching by means of air produces the best result; the linseed oil is least altered, it is somewhat thickened and becomes rancid, but the process is very lengthy and demands an exposure of months. The best process is as follows: 100 parts (by weight) of linseed oil are washed with 100 parts of water and 2 parts of strong hydrochloric acid in a vessel of suitable size, of wood or iron coated with lead, which possesses a tap at the height of the level of the water. The mixture is well stirred for 24 hours, it is then allowed to rest until the oil has separated and become completely clear, when it is drawn off, the water removed, and the oil brought back into the vessel, where it is thoroughly washed with water and then again allowed to clear. This purified oil is much more suitable for bleaching than oil which has not been treated. It is brought into shallow wooden tanks lined with sheet zinc or lead; the depth of the oil should not exceed 10 cms. These tanks must be placed in some position where the sun can shine upon them during the whole day. The best position is on a flat roof under a glass cover, so that rain, dew, and dust cannot fall into the oil. Such an arrangement is naturally not cheap, and can only be profitable when a large quantity of bleached oil is produced. With favourable weather it may be assumed that the oil will be completely bleached and white in 4 weeks.

The common practice of exposing oil in round glass bottles, indeed produces the same result, but after a very long time, months, or even a year, if the light is insufficient, may pass before a proper result is obtained. But also in this case, if the oil be exposed in thin layers to the action of light, it is bleached colourless in a much shorter time. This exposure in thin layers is possible if flat glass bottles are used to contain the oil; they can always be obtained. For experience has shown that, in order to obtain powerful bleaching action, the liquid to be bleached must not simply be exposed to the direct rays of the sun, but that the layers should be so placed that the rays must pass through them. If the oil be exposed in quite thin layers under glass to the action of light, a very appreciable loss of colour can be observed even after several days.

Bleaching by means of sodium peroxide is accomplished by thoroughly working a solution of 5 parts in 100 parts of water with 100 parts of oil (by weight) at a temperature of 25° to 30° C. for at least 2 days. The oil is allowed to separate; it is well washed, first with hot, then with cold water. The method does not give particularly good results.

Another process is as follows:—In a lead or copper vessel, provided with a good stirring apparatus, 200 kilos. of raw linseed oil, at a temperature not exceeding 21° C., are slowly mixed with 0·5 kilo. of sulphuric acid (specific gravity, 1·75), whilst stirring. After well mixing, which requires about 15 minutes, 0·5 kilo. of sodium peroxide is very slowly added. The addition should last at least 60 minutes, and the stirrer should be kept in rapid motion. When all has been added, stirring is continued for at least 12 hours, the oil allowed to stand, and when clear washed first with warm and finally with boiling water.

Bleaching by means of chlorine.—This bleaching agent gives, after light, the best results, it is also the cheapest, and the bleached oil can very readily be washed and obtained completely neutral, since only salts soluble in water are present. In bleaching with chlorine, 5 parts (by weight) of bleaching powder (as fresh as possible) are well mixed with 20 parts of water, then 0·5 part of soda (carbonate) is added, and the solution filtered so that it is quite clear. Next, 25 kilos. of linseed oil, 500 grms. of strong hydrochloric acid, and 5 kilos. of water are well mixed for 1 to 2 hours, and the filtered bleaching powder solution added. The two liquids are repeatedly stirred thoroughly together during 5 to 6 hours, when the mixture is allowed to rest, the clear oil drawn off and repeatedly washed with clean water. The oil is almost colourless and must have a perfectly neutral reaction. It is absolutely necessary to prepare the clear solution of bleaching powder, for if an unfiltered solution were used the oil would be partially saponified, and would then be regained only by a very complicated process. The method of bleaching by means of hydrochloric acid and pyrolusite, which produce free chlorine, is impracticable, since the pyrolusite, on account of its insolubility, is never completely used up, so that large quantities would be required to produce any considerable bleaching action.

Bleaching by means of hydrogen peroxide.—Pure hydrogen peroxide is a syrupy liquid, colourless, and quite transparent, with an unpleasant bitter and biting taste; when brought upon the skin it produces violent irritation and white spots. The commercial substance contains about 1½ volumes of available oxygen, corresponding to about 0·5 per cent. of H_2O_2. To preserve it, hydrochloric or sulphuric acid is added, to which regard must be had in certain cases.

In order to bleach linseed oil by means of hydrogen peroxide, it is mixed with 5 per cent. of the solution and then with sufficient ammonia to produce a distinct smell of that substance. Without the addition of ammonia or alkalis the action of hydrogen peroxide may be protracted for several days, without any visible action upon the oil. There is no difficulty in brightening the oil, and loss due to emulsions is not to be feared, as is the case in so many bleaching methods. The process must thus be regarded as very convenient.

Bleaching by means of potassium permanganate.—Potassium permanganate is a very powerful bleaching agent; it possesses the advantage of not spoiling the oil in any way, but, on the contrary, makes it dry more rapidly. The process is very simple, and if the correct proportions are observed the formation of emulsions is small. A solution of 16 parts (by weight) of potassium permanganate in 32 parts of water is prepared, 5 parts (by weight) of this solution are mixed with 100 parts of linseed oil; 25 parts of water and 1 part of strong hydrochloric acid are then added and the whole well stirred. The mixture must be stirred up repeatedly during 24 hours, and left at rest when all violet or brown coloration has disappeared and the mixture has become yellow. After several days the oil will have entirely separated from the bleaching liquid, it is then drawn off. The oil is almost colourless; when necessary, it can be again subjected to the bleaching process in order to make it quite colourless.

Bleaching by means of potassium bichromate.—Linseed oil is bleached by means of potassium bichromate in a wooden tub, in which it is heated by direct steam to 31° to 32° C. A solution of 3 per cent. of potassium bichromate, dissolved in 5 times its quantity of water, is run in and the mixture well stirred by means of a stirrer, after the addition of strong hydrochloric acid equal in quantity to 1 per cent. of the oil. The decolorisation begins quickly, and is complete in 10 to 15 minutes. The oil is left at rest for a long time, in order that the aqueous solution of potassium and chromium chlorides may settle. This solution is drawn off by means of a tap, with which the vessel is provided, and the oil washed with hot water to remove the chromium salts and any hydrochloric acid which may be present. The oil is again left at rest, and when clear is drawn off. Sodium bichromate may well be used in place of the potassium salt; it is cheaper and also more easily soluble in water, so that the bleaching solution need not be used boiling, as must be the case with potassium bichromate.

Bleaching by means of ozone.—The linseed oil to be bleached is brought in contact with ozone in shallow boxes, which are best lined with sheet lead, and through which air richly charged with ozone is allowed to pass. The ozonised air may also be aspirated through the oil, which is to be thickened and bleached, in tall, narrow vessels, and the oil then exposed to the action of light and air in shallow vessels for several days. When treated in this manner linseed oil bleaches in a few days, at the same time absorbing so much oxygen in the short period that it thickens to a viscous oil.

CHAPTER IV

THE MANUFACTURE OF BOILED OIL—THE PREPARATION OF DRYING OILS FOR USE IN THE GRINDING OF PAINTS AND ARTISTS' COLOURS AND IN THE MANUFACTURE OF VARNISHES, BY HEATING (BOILING) OVER A FIRE OR BY STEAM, BY THE COLD PROCESS, BY THE ACTION OF AIR, AND BY MEANS OF THE ELECTRIC CURRENT

THE use of drying oils, especially of linseed, poppy, and walnut oils, in conjunction with a pigment, either with or without substances which increase the drying power of the oil, is of comparatively ancient date. The discovery that linseed oil could be converted into a "varnish" by means of litharge, is attributed to an Augustine monk. In books of the end of the sixteenth century we find directions for preparing a good drying "varnish" by means of red lead, litharge, bread, garlic, etc. These ancient boiled oils were thick and dark brown and had a strong smell, since, in order to make a good "varnish," the oil had to be heated for a long time and with a large quantity of drier. Such boiled oils have lasted almost to the present time; twenty years ago there were consumers who regarded boiled oil as good only when it possessed the properties mentioned above. These boiled oils were made in a very primitive manner: for ordinary use they were generally made by heating the oil up to a high temperature, yet not so high that decomposition occurred. Pieces of bread and onions were then thrown in, which were allowed to become thoroughly brown, when they were eaten with relish. The driers were next added, and the oil heated up to over 300° C. Others added the driers when heating was commenced, tied up in a linen bag, which was hung in the oil, so that only oxygen could pass into the oil. Others, again, added the driers in very small quantities whilst the oil was being heated, allowing each portion to "dissolve" before more was added. The vessels in which the oil was boiled differed as much in shape as in size. Some believed that the heated oil must present a large surface to the air, and therefore used wide shallow vessels. Others boiled in deep pans and stills, so that no air could enter to the oil.

At the present time all this has been changed; the manufacture of boiled oil, which was formerly carried on by the consumers,—artists and painters,—is now in the hands of manufacturers, who concern themselves exclusively with it or combine it with the production of the oil, the manufacture of paints or of varnishes. As a rule the apparatus employed corresponds with the present condition of technology, but in many cases boiled oil is still made by the older processes.

The drying oils, as they are obtained by expression or extraction, do not quite answer to the demands made of an oil to be used in conjunction with pigments. For although they do dry without any additions, and relatively more quickly in combination with certain pigments (white lead, red lead), yet the duration of drying is much too long.

Linseed oil requires	3–7 days.
Poppy ,, ...	2–5 ,,
Walnut ,, ...	2–5 ,,
Candle-nut ,, ...	1–2 ,,
Chinese wood oil requires . . .	¾–1 ,,

The oils have therefore to be subjected to certain processes of preparation, which are—

(1) Heating to a more or less high temperature with certain substance (metallic oxides).

(2) Blowing air through the oil.

(3) The electric current or ozone is allowed to act on the oil.

The older views, upon the manner in which driers acted upon drying oils, were essentially that a certain quantity of oxygen must combine with the oil, which oxygen originated in metallic oxides decomposed in the hot oil, or in atmospheric air or pure oxygen; thus that oxygen must be absorbed in order to increase the drying power. According to Mulder, for the formation of boiled oil "a high temperature and the addition of oxygen, whether obtained by the action of air or by the reduction of added metallic oxides rich in oxygen, are absolutely necessary." In accordance with these views, the preparation of boiled oil produces the following changes:—

"(1) A portion of the linolic acid and other fatty acids of linseed oil is set free.

"(2) In the presence of bases the fatty acids form salts.

"(3) The formation of the anhydride of linolic acid is effected or initiated.

"(4) Two or three of these processes take place at the same time.

"Without any treatment, every drying oil produces, though slowly, the leathery linoxyn and free fatty acids, which are always formed in the preparation of the oil in larger or smaller quantity, according to the duration of the boiling."

Such was the condition in which our knowledge of the formation of boiled oil was left by Mulder's researches. It was thought that boiled oil could not be made without adhering to the above principles. These principles were advanced in the author's previous work on this subject. Since then there has been a great change in our knowledge; by various researches it has been shown that the preparation of the drying oils, in order to impart to them great drying power, does not depend upon the addition of oxygen in any manner, but on the introduction of such substances as are capable "*of absorbing oxygen from the air, and of transferring this oxygen to the linseed oil when it is spread out in a thin layer*," so that the oil uses the oxygen of the air for its own oxidation. This absorption of oxygen from the air by the substance contained in the oil, and its transference to the oil, proceed until the coating is quite dry (in thicker layers until very viscid). Full details of the researches on this subject have been given in Chapter I.; the results are mentioned here since they are absolutely necessary to a general understanding of the subject.

The methods employed in the practical preparation of boiled oil had been actually based upon this principle before it was proved. Many years ago a good drying linseed oil was made by the addition of liquid driers to raw linseed oil, without the application of heat; the product was not, however, regarded as equal to boiled linseed oil. With the introduction of the metallic resinates a further advance was made; they were long used in the preparation of boiled oil, without the application of heat or at a very moderate temperature, before it was made certain by scientific research that drying takes place simply through the transference of oxygen.

According to Fehr, the alterations which linseed oil undergoes in boiling are loss of mucilage and initiation of the oxidation.

(1) *The loss of mucilage* is regarded by Fehr as the most important result of boiling oil. In this connection he says:

"If a small quantity (a pinch) of litharge be introduced into linseed oil containing mucilage, the mixture frequently shaken and left at rest for several days for the oil to brighten, there will be found upon the bottom of the vessel, in addition to the uncombined oxide, a slimy deposit, which consists of the mixture of gum and albumin, generally termed mucilage. The latter forms the great impediment to the drying, or more properly, the hardening, of linseed oil, so that the quality of the oil depends on the larger or smaller quantity of this mucilage. The mucilage is a soft mass, soluble in oil, which, when present in large amount in linseed oil, makes it thick and viscous. The first requirement which should be demanded of boiled oil is that it should be sufficiently fluid, so that in this respect it stands between raw and refined oil. The clear liquid above the deposit, although not quite free from mucilage, has still lost a considerable proportion. It will be found to have become considerably more fluid, and hence that it will dry more quickly than the raw oil."

The mucilage may also be more successfully removed by other metallic oxides, but the removal is more rapidly and completely accomplished by the action of heat, on which account the oil is "boiled," as it is falsely expressed, in the manufacture. This description is false, since the boiling point of linseed oil lies above 300° C.; the formation of bubbles in the oil, which is erroneously regarded as boiling, begins at 100° C., and is only a result of the evaporation of the water in the oil.

What is then the action of heat alone and of litharge when linseed oil is heated? As has been said, the evaporation of water commences at 100° C. If the temperature be further raised, at about 150° C. the yellow colouring matter begins to burn, and is completely destroyed at 180° C., when the oil is decolorised and has acquired a pale greenish-yellow colour; at this temperature the consistency has not changed. The alteration does not commence below 300° C., thus it occurs simultaneously with the formation of vapour, *i.e.* the decomposition of the oil into its constituents. As is the case with wood, the hydrogen here also burns more rapidly than the carbon, the oil becomes richer in carbon, turns brown, thickens, and acquires greater drying power. The vapours evolved are principally hydrocarbons; they readily ignite. In boiling linseed oil which is intended for use in paints, the boiling point of the oil should never be reached, in order that the oil may not thicken, which, apart from other disadvantages, would considerably increase the labour of the painter. The temperature should be raised to 175° to 180° C., in order that the water may be removed and the oil made as colourless as possible. It is one of the first requirements of boiled oil that it shall not impair the shade of pale colours.

If now litharge be introduced into linseed oil heated to this temperature, the mucilage separates almost at once. The oil retains its fluid nature if now removed from the fire; but if kept with the litharge for some time at the high temperature it becomes brown and thick, although the temperature is far below the boiling point. In this case the thickening is due to the litharge, which disappears as such more or less from the oil; it combines with the acids of the oil and a thickish liquid is obtained which dries very rapidly, and thus is described as boiled oil, though in reality it does not deserve the name, but rather should be ranked with the "plaster varnishes," which are known to have little durability. On the contrary, that boiled oil offers the greatest promise of durability in which the oil has been kept as far as possible in its natural condition during the operation of removing the mucilage; this is, however, not the case with thick boiled oils.

Thus it would be proper to require of boiled oil that it should be thin, like the oil from which mucilage has been removed.

(2) *Initiation of the oxidation of the oil.*—It is generally thought that, in boiling oil with metallic oxides, the oxidation is commenced by the oxygen of the oxide and carried forward to a certain point. This belief is founded on the fact that it is oxygen which causes boiled oil to dry, and confirmed by the rapid drying of oil in contact with lead pigments which contain oxygen, as opposed to the slow drying of oil in contact with pigments, such as the soot blacks, which contain no oxygen. Fehr regards this view as incorrect, at least in so far as the oxygen of the oxide is supposed to effect the oxidation of the oil. If this were the case the oxygen would be separated from the oxide, so that metallic lead would form a deposit in lead boiled oils. As a rule this does not occur, and in the exceptional cases in which it is observed it signifies simply that the oxygen has separated from the lead, but not that it has combined with the oil. That view is also opposed to the experience that few metallic oxides produce boiled oil drying as quickly as is obtained with manganese borate, and further, that zinc oxide, in spite of its high percentage of oxygen, is a pigment in contact with which oils dry slowly. In order to decide this question, Fehr passed a current of oxygen for a long time through raw linseed oil. The drying properties remained unaltered. Thus it is to be concluded that in boiling oil the oxygen of the metallic oxide plays no part. The oxides, however, unite directly with the oil and cause the separation of the mucilage from the oil, which process is sufficient to make the oil absorb oxygen from the air more rapidly. The oxidation which the oil undergoes in increased measure at the temperature of boiling is caused solely by the oxygen of the air and not by the metallic oxides.

In addition to litharge, several manganese compounds are used in boiling oil, such as manganic hydrate, and especially manganous borate. These substances effect the separation of mucilage from the oil to the same degree as litharge, and have the advantage over the latter that they do not thicken the oil. Since, however, there is danger of producing dark boiled oil, the temperature should not exceed 175° C., at which manganous borate acts upon the oil and produces a thin pale boiled oil with a somewhat greenish tinge. This oil is completely indifferent to sulphur compounds, and hence may be used with all pigments, and for both indoor and outdoor work.

Both in boiling and later, when the oil dries, the oxidation is caused by the oxygen of the air. It is completed more readily in oils from which the mucilage has separated, and is accelerated by heating the oil. Too long or too violent heating causes excessive oxidation, and hence thickening, also under certain circumstances it darkens the oil.

Success in the production of boiled oil depends, apart from the first requisite—raw oil of good quality, upon heating the oil to the correct temperature, which is readily obtained if a thermometer is used.

Livache gives the following as the conditions upon which the production of a good boiled oil depends :—

(1) The quantity of the drier or driers.

(2) The temperature:

(3) The duration of boiling.

In boiling linseed oil over an open fire the quantity of driers varies from 2 to 4 per cent. of lead compounds and from 1 to 2 per cent. of manganic hydrate or manganous borate. If the deposit be examined at the conclusion of the operation a slimy mass will be found, and in the case of lead compounds a notable quantity of metallic lead, whilst the boiled oil contains only about 1 per cent. of lead oxide. Hence we may conclude that

(1) The oxidisable constituents of the oil have retained a certain quantity of lead oxide.

(2) A certain quantity of lead oxide is precipitated in the form of insoluble lead salts.

(3) A portion of the lead oxide has formed an insoluble compound with the oil. This compound is due to the ready saponification of certain glycerides in the oil, whereby a certain quantity of glycerin is set free, corresponding to the saponified oil.

If a manganese salt be employed instead of the lead salt, the boiled oil contains about 0·5 per cent. of manganic oxide.

In practice the suitable temperature for boiling is regarded as 210° to 228° C., and it is easy to give the reasons.

In the first place, experience shows that when the oil is heated it begins to decompose at 230° to 236° C., and gives off volatile products, whilst fatty acids are formed which injure the oxidised oil and require a long time to dry. In order to obtain a fluid product which has not perceptibly thickened, the temperature should not exceed 230° C. In the saponification which occurs a corresponding quantity of glycerin is set free. If this remained in the oil the dry layers would never completely harden, but would remain tacky. The glycerin must be removed, and this is accomplished by boiling for a long time, since part of the glycerin is evaporated at 225° C., together with certain volatile materials which are evolved in small quantities from the oil during boiling; the temperature has little influence upon the distillation products of glycerin. Another part of the glycerin is decomposed, either by the long continued heating or, as Hartley has shown, by the presence of metallic lead (which has been shown to be present at the end of the operation), into volatile products such as acroleïn, acrylic, formic, and acetic acids. The largest quantity, acting upon the triglycerides, produces diglycerides, the complete oxidation of which follows, and a perfectly dry skin is formed when this last reaction takes place at a temperature of 200° C. In order to obtain good boiled oil, the temperature should not exceed 230° C., but it must rise above 200° C. In practice, as we have said, it is maintained between 220° and 228° C.

The duration of boiling is from 3 to 6 hours, according to the shape of the apparatus, the intensity of the fire, and the quantity of oil under treatment. It is necessary to boil until the above-mentioned reactions have occurred. It has, however, been shown that the quality of boiled oil is better the longer it has been boiled. A polymerisation of the different glycerides of the oil occurs, producing an oil which oxidises much more rapidly, and when dry has greater elasticity. This is the reason why, in England, instead of boiling oil rapidly and causing it to "break," as in France, the oil is very slowly boiled, and the boiling process protracted for several days and even for a week.

In working with steam or superheated air the temperature is maintained between 125° and 130° C. Steam at a pressure of 4 to 5 atmospheres becomes reduced to about 2 atmospheres pressure in the steam coil or jacket of the pan. It will readily be understood that it is difficult, in view of safety and economy, to heat steam to such a temperature that it indicates 200° C. in a coil or jacket, for about 15 atmospheres pressure are required. At a lower temperature the glycerin formed by partial saponification remains in the oil in large quantity and prevents the formation of a coating as hard as is produced by fire boiled oil. However, oil boiled by means of steam has the great advantage that it does not become dark, hence manganic oxide can be used in larger quantity, so that an energetic oxidation is produced, which counteracts the unfavourable action of the glycerin. Direct oxidation may also be produced by sending a current of air through the oil. The glycerin may also be removed by sprinkling water upon the hot oil. It will thus be understood that fire boiled oil is often preferred to oil boiled by means of steam; prolonged boiling may, however, avoid or quite remove the defects of the latter.

It is shown by these statements of Fehr and Livache that heating to 220° C. is absolutely necessary, on the one hand, to separate the mucilage, and on the other, to produce an oil which shall subsequently dry to a hard layer. The author agrees with these views only in so far as relates to the separation of the mucilage from the oil, and when a pale oil was in question would feel no anxiety concerning the good, hard drying of an oil which had only been heated to 130° to 135° C. It will be seen later that it is possible, with suitable driers, to produce a good and hard drying boiled oil quite without the application of heat. The substances which have been used or proposed for use as driers will next be described.

The Driers Used in Boiling Linseed Oil

In order to convert raw into boiled linseed oil, certain substances, the compounds of particular metals, are introduced into the oil. They have the property of absorbing oxygen from the air and transferring it to the oil, by which the latter is made capable of yielding a hard elastic layer.

Numerous substances have been used or proposed for this purpose during a long series of years. A list of this is given for the sake of completeness; their usefulness will be subsequently discussed:—

Alum.
Antimony oxide.
Boric acid.
Bread.
Calcium plumbate.
Copper linoleate.
Copper resinate.
Copper salts (nitrate, sulphate, etc.).
Dogs' excrement.
Ferric oxide.
Ferrous hydroxide.
Flake white (white lead).
Garlic.
Gypsum.
Lead, the granulated metal.
Lead acetate.
Lead borate.
Lead carbonate and white lead.
Lead-lime (lead peroxide and lime).
Lead linoleate.
Lead manganate.
Lead-manganese linoleate.
Lead-manganese resinate.
Lead oxide, prepared.
Lead oxides, litharge (silver or gold), red lead.
Lead peroxide.
Lead, precipitated (by the action of metallic zinc on lead acetate solution).
Lead resinate.
Lead suboxide.
Lead tartrate.
Manganese acetate.
Manganese benzoate.
Manganese borate.
Manganese carbonate.
Manganese chromate.
Manganese citrate.
Manganese formate.
Manganese linoleate.
Manganese nitrate.
Manganese oleate.
Manganese oxalate.
Manganese peroxide.
Manganese peroxide, artificial (containing 70 to 75 per cent. of MnO_2, 10 to 15 per cent. of MnO).
Manganese preparation (resinate).
Manganese resinate.
Manganese succinate.
Manganese sulphate.
Manganese sulphite.
Manganese tartrate.
Manganic chloride.
Manganic oxide, crystallised (braunite).
Manganous hydroxide.
Mercuric oxide, red.
Nitric acid.
Onions.
Pumice.
Pyrolusite.
Shale, brown shale.
Tin, the granulated metal.
Umber.
Verdigris.
Vermilion.
Zinc, the granulated metal.
Zinc oxide.
Zinc sulphate (zinc or white vitriol).

Preparations of unknown or inexactly known composition:—

Boiled oil preparation.
Manganese oxide in lumps.
Prepared drier.
Ozonolin, a substitute for litharge.
Resinate extract.
Siccative extract.
Siccative powder.
Universal siccative (containing lead and manganese).
Varnish preparation (manganese resinate).

From these alleged driers the following may be separated as quite inactive and therefore useless:—Alum, antimony oxide (decreases drying power), boric acid, bread (used to remove "fat" from linseed oil), copper resinate, linoleate, and sulphate (which have again recently been alleged to cause drying), dogs' excrement, ferric oxide, ferrous hydroxide, garlic, gypsum (only removes water), manganous chloride, mercuric oxide (red), nitric acid, onions, shale, tin (granulated metal), umber, vermilion, zinc (granulated metal), zinc oxide. When the remaining substances are examined, after the removal of the useless "driers," it is seen that they are lead and manganese compounds and zinc sulphate; the use of the last named is not general, and its usefulness is often denied.

[TABLE.

SUMMARY OF MANGANESE COMPOUNDS FOR OIL BOILING (USED OR PROPOSED).

Compound.	Formula.	Molecular Weight.	Theoretical percentage of Manganese.	Average percentage of Manganese in Commercial Wares.	Colour.	Temperature at which the Substances yield a Good Drying Oil; Duration of Action, 2 hours.
Pyrolusite	MnO_2	87	63·22	30–60, 55	Black.	About 250° C.
Manganic hydroxide . . .	$Mn_2O_3.H_2O$	176	62·50	35–55	...	170°–200° C. More safely, 200°–220° C.
Manganese peroxide, hydrated .	$MnO_2.H_2O$	105	52·38	35–55	...	
Manganous hydroxide . . .	$MnO.H_2O$	89	61·80	35–55	...	
Manganese chloride . . .	$MnCl_2.4\ H_2O$	198	27·78	About 27	Rose.	About 250° C.
,, sulphate . . .	$MnSO_4.H_2O$	169	32·51	27–31·5	White.	Above 250° C.
,, nitrate . . .	$Mn(NO_3)_2.6\ H_2O$	287	19·16	19–20	Rose.	150°–170° C.
,, chromate . . .	$MnO.MnCrO_4.H_2O$	260	42·30	38–45	Black.	200° C.
,, borate . . .	...	...	...	5–22, 15	White.	...
,, carbonate . . .	$MnCO_3.H_2O$	133	41·35	41–42	Pale brown.	About 280° C.
,, oxalate . . .	$MnC_2O_4.2\ H_2O$	179	30·37	About 30	White.	Above 280° C.
,, succinate . . .	$MnC_4H_4O_4.9\ H_2O$	243	22·52	22·5	Pale salmon.	About 270° C.
,, citrate . . .	$Mn_3(C_6H_5O_7)_2.9\ H_2O$	705	23·40	22·0	White.	,, 270° C.
,, tartrate . . .	$MnC_4H_4O_6.2\ H_2O$	239	23·01	23·0	Pale salmon.	Above 270° C.
,, benzoate . . .	$Mn(C_7H_5O_2)_2.4\ H_2O$	369	14·90	14·8	,,	About 200° C.
,, formate . . .	$Mn(CHO_2)_2.2\ H_2O$	181	30·38	29·7	Pale violet.	,, 270° C.
,, acetate . . .	$Mn(C_2H_3O_2)_2.4\ H_2O$	245	22·45	About 22	Rose.	120° C.

VARIOUS DRIERS, THEIR THEORETICAL AND ACTUAL PERCENTAGE OF METAL, AND THE MINIMUM TEMPERATURE AT WHICH THEY PRODUCE BOILED OIL.

Drier.	Theoretical Percentage of Metal.	Average Percentage of Metal in the Commercial Substance.	Minimum Temperature for the Production of Boiled Oil.
Pyrolusite . . .	63·2	(30–60), 55	About 250° C.
Manganic hydroxide .	62·5	45–50	170–220° C. More certainly at 200°–220° C. Eventually in the cold.
Manganese dioxide, hydrated (artificial pyrolusite) . . .	52·4	45–50	
Litharge	92·8	About 93	
Red lead	91·2	,, 91	
Manganese borate . .	...	(5–22), 15	
,, acetate, crystallised .	22·5	About 22	
Lead acetate, crystallised	54·6	,, 54	
Manganese oxalate . .	30·4	,, 30	
,, carbonate .	41·4	,, 41	Above 280° C.
,, resinate, precipitated .	About 7·7	About 7 soluble	Without rise of temperature.
,, resinate, fused	,, 5·3	2·5–4·5 ,,	
,, linoleate .	,, 8·9	About 9 ,,	
Lead-manganese resinate, fused .	...	...	
,, linoleate	...	...	
Powdered siccative . .	...	0·5–3	
Liquid ,, . .	...	...	

The useful driers, which have been mentioned, may be divided—

(1) Into those which have been used since the first manufacture of boiled oil, and have been somewhat more extensively employed with the advance in the preparation of new compounds of lead and manganese, viz. the oxides and salts of these metals with inorganic acids and organic acids of low molecular weight, such as pyrolusite, artificial oxides of manganese, litharge, red lead, manganese borate, acetate and carbonate, lead acetate, etc. Of these, lead acetate, litharge, red lead, pyrolusite, manganese borate, manganic oxide, manganic hydroxide and peroxide have been in practical use until quite

recently. The other compounds, especially the different manganese salts, have had occasional and not very successful employment.

(2) Into those which have become known during the last fifteen years—lead and manganese salts of the acids of high molecular weight derived from animal and vegetable fats and resins—the so-called soluble driers or "soluble siccatives," as they have been unfortunately called.

The essential and characteristic difference between the two kinds of driers is, as Weger has said, that the members of the second group dissolve readily and in large quantity in linseed oil (also in turpentine and benzine) at the ordinary temperature, or slightly above it. The members of the first group, on the other hand, are insoluble in turpentine, and generally require a higher temperature to be dissolved by linseed oil.

The great variety of fats and oils offers an extensive choice for the preparation of the driers of the second class. According to Weger, all the possible lead and manganese salts may be made and used with tolerably equal effect. The linoleates and resinates have alone been introduced; this is natural, since linseed oil and resins are the fundamental constituents of boiled oil and varnish respectively, and also because they are practically the cheapest materials. An oleate (the linoleates are often called oleates) or a palmitate may be occasionally used, but the quantity is trifling as compared with the resinates and linoleates. The acid, which is combined with lead or manganese, has doubtless some influence upon certain properties of the boiled oil, but it is *à priori* to be expected that it is without influence on the rapidity of the drying, and that only the quantity of lead or manganese influences the transference of oxygen. This expectation was experimentally confirmed for the lead and manganese linoleates and resinates respectively. In the case of the manganese compounds the experiments were not quite conclusive, the linoleate oils appeared to dry more rapidly than the resinate oils.

The commercial lead and manganese salts of the oil-acids and resins are the following:—Lead resinate, lead oleate, resinate of lead and manganese, oleate of lead and manganese, manganese resinate (fused and precipitated), manganese oleate (lead or manganese oleate or resinate). These are tolerably complicated compounds, the chemical constitution of which is not known with the same accuracy as that of the salts of acetic or oxalic acid.

The compounds mentioned above contain the following quantities of active metal:—

Manganous linoleate,	about	8·9	per cent. of	manganese.
Manganic linoleate,	„	6·1	„	„
Lead linoleate,	„	26·9	„	lead.
Manganous resinate,	„	7·7	„	manganese.
Manganic „	„	5·3	„	„
Lead „	„	24·0	„	lead.

These figures may vary in the case of resinates by about 5 per cent., in the case of linoleates by 2 per cent. above or below. In consequence of the method of preparing these substances, especially of those obtained by fusion, complications arise which make the value of these figures very problematical. Deep-seated decompositions are produced at the high temperature; what becomes of the glycerides is doubtful. In the case of manganese preparations, reduction and oxidation may occur. It is not known whether the product obtained from rosin and manganic oxide is really a manganic salt, or whether it is not partially manganous salt. The principal difficulty is, however, that under the usual conditions no absolute union of base and acid or glyceride occurs, but rather, in spite of an excess of the former, uncombined acid or unattacked glyceride remains. On the other hand, under certain conditions, as when the process is "forced," more metal can be dissolved by the linseed oil, and apparently also by the rosin, than corresponds with the extreme limits of the figures just given. Weger found 11 per cent. of soluble manganese in a fused manganous linoleate, and more than 30 per cent. of soluble lead in a fused lead linoleate. It might readily be supposed that the glycerin residue had combined with the excess of lead or manganese; this is not impossible in the case of lead. It is, however, very improbable in the case of manganese, since free glycerin is only able to dissolve slight traces of manganous or manganic oxide even at high temperatures. Experiments made with linoleïc acid free from glycerin seem to decide against the above hypothesis. It has not yet been shown that the products containing an abnormal quantity of metal are quite free from uncombined rosin or linseed oil.

For the purposes of oil boiling, it is now important to recognise the value of the driers in regard to their condition and purity. The lead oxides of commerce, red lead and litharge, are generally produced in a commercially pure condition, and in this form they suffice. Red lead rarely corresponds to the formula Pb_3O_4, it generally contains somewhat less oxygen; it contains 17·5 to 33·6 per cent. of peroxide and 65·0 to 75·1 per cent. of monoxide. Red lead is adulterated with brick dust, red ochre, ferric oxide, barytes, gypsum, lead sulphate, clay, and sand, more rarely with white lead, zinc white, calcium or barium carbonate, etc., substances which are inactive. Pure litharge contains 92·83 per cent. of lead and 7·17 per cent. of oxygen; the impurities are the oxides of copper, iron and antimony, lead, lead suboxide, red lead, also chalk, silica, silicates (clay), carbonic acid, more rarely the oxides of silver, bismuth, tin and zinc. Occasionally brick dust, red ochre, and ferric oxide are fraudulently added to litharge, decreasing its value. The remaining lead compounds included in the first group, when commercially pure, answer to the requirements. The same is the case with the manganese compounds; still, it should be observed that adulteration with zinc white is common.

In the compounds of the acids of rosin and of the oils, the soluble metal, *combined* with the rosin or the oil-acids, is alone of importance. The compounds suspended in the rosin or oil (MnO_2, Mn_2O_3, $MnCO_3$, PbO, etc.), which occur in many commercial products, do not enter into reaction when the drier is dissolved at the ordinary temperature or at a gentle heat. They are also harmful, since they make the oil turbid. On the other hand, a large excess of rosin or oil is to be avoided, since it would diminish the percentage of the active constituent. Excess of linseed oil in the drier would naturally not be harmful; a large excess of rosin is strictly to be avoided. If we take the case in which the resinate drier contains 50 per cent. of free rosin, the oil would contain at the most 1·5 per cent., since more than 3 per cent. of drier is very rarely used—thus at the worst a very inconsiderable quantity. Attempts to restrict the quantity of free acid are generally at the expense of the complete solubility of the drier and its pale colour, and at the same time frequently no improvement results. All fused resinates contain free rosin or uncombined mineral matter, and very often both at the same time. The free rosin in a drier has a considerable influence on its easy solubility at low temperatures; a fused manganese resinate, containing 5 per cent. of soluble manganese, would *à priori* be preferred to a resinate containing only 4 per cent., and in certain cases is preferred in practice. In other cases it is quite useless, whilst the 4 per cent. resinate can be employed at a pinch, but a 3 per cent. resinate would be most suitable.

As regards adulteration of this class of driers, it does not occur in the case of resinates. Those who do not know the requirements of the manufacturer might regard free rosin as an adulterant added to decrease the cost; such an addition would not, however, perceptibly diminish the cost. Linoleates are commonly adulterated; the adulterant in one case was found to be, not free rosin, but resinate of lime (50 per cent.). Manganese linoleate may contain small quantities of calcium linoleate in consequence of the material used in its preparation; small quantities of calcium resinate would not be detrimental, but large quantities should not occur. To test for this adulterant, the acids are separated from the suspected substance and examined in Wollny's butyro-refractometer, or, in case this expensive instrument is not at hand, the resin and fatty acids are separated by Twitchell's method.

Bornträger of Hanover has practically examined various driers for linseed oil. He finds that chrome red, obtained by boiling lead chromate with caustic potash, and also lead chromate, make linseed oil dry in 24 hours. Ignited lead chromate, a pale brown powder, apparently containing lead peroxide, dries with linseed oil in 12 hours. Lead humate, a black powder, effects drying in 24 hours; ignited lead humate and lead suboxide, in 24 hours, very hard; lead oxide, heated in the oxidising flame, in 12 hours; ignited zinc humate (zinc white), in 15 hours; Chinese blue, in 12 hours; basic zinc chromate, in 24 hours; basic lead chromate, precipitated by sulphuric acid, a cinnamon-coloured powder, in 24 hours. The lead chromates, pure and ignited, dry best, as also ignited lead and zinc humates.

Unfortunately, in the communication the manner of treating the linseed oil is not mentioned, whether in the hot or cold way, and also the quantities used, which would be of importance. The author doubts whether lead chromates would effect any increase in the drying capacity; in the case of Chinese blue, it is quite out of the question, it is well known in practice that all the iron-cyanogen pigments rather hinder than accelerate drying, and that they are generally used with a large quantity of liquid drier. The success obtained with ignited lead chromate was perhaps due to the presence of finely divided metallic lead. The action of lead humate may be ascribed to a similar reduction.

It may now be regarded as decided that the lead and manganese compounds cause the most energetic transference of oxygen to linseed oil, and that they alone find application as effective driers. Many compounds of these metals have been proposed from time to time, which probably in most cases, if not always, are inferior to the resinates.

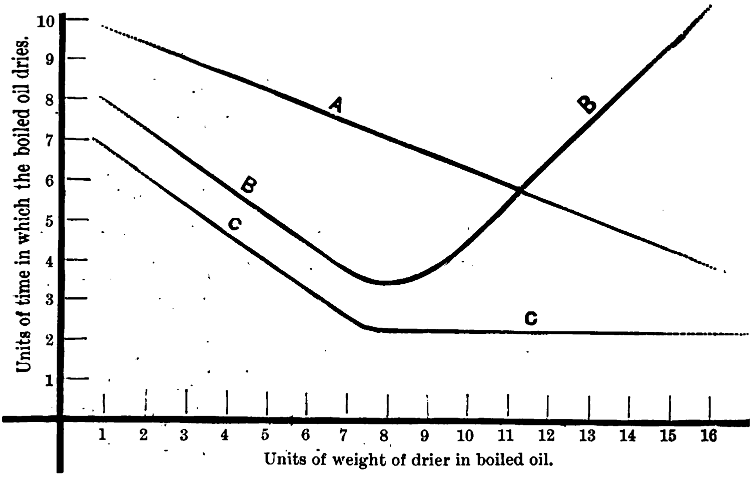

Fig. 17.—Curves showing Connection between Proportion of Driers in Boiled Linseed Oil and Time of Drying.

In regard to the relation between the drying power, or the time required for drying, and the quantity of lead or manganese, or, as it is better expressed, the quantity of drier, Weger makes the following statement:—If a small quantity of a solid manganese compound, corresponding to about 0·02 per cent. of manganese, be dissolved in raw linseed oil, it dries perceptibly more quickly than without manganese. If the quantity of manganese be increased to 0·05 per cent. the drying action is already noticeable after 12 to 15 hours. If the quantities of metal dissolved in boiled oil be taken as abscissæ, the times of drying as ordinates, any one of the curves in Fig. 17 may theoretically represent the relation.

Weger's experiments show that the curve C really represents the connection. The experiments were performed with commercial fused manganese resinate, manganese linoleate, manganic hydroxide, and lead-manganese resinate. In all such experiments it is necessary to estimate the quantity of metal (or driers) actually contained in the oil. In order to obtain distinct differences in the times of drying, it is necessary to work with large quantities of driers, which in many cases are not permanently retained by the oil. Weger comes to the conclusion that, as the quantity of drier increases, the drying power first becomes greater and then remains constant; the curve therefore falls at first and then runs horizontally. The minimum time of drying is obtained with about 0·2 per cent. of manganese, which large quantity is therefore excessive in practice. With lead-manganese oils the quantity of drier may be greater; the minimum time of drying is attained with about 0·6 per cent. of metal in the proportion of 5 Pb to 1 Mn. The belief, not seldom expressed, that too much drier reduces the drying power, originates from observations made on linseed oil mixed with liquid driers; excessive quantities of driers do not remain permanently in the oil, but separate.

F. W. Thorpe made a number of experiments in order to elucidate the influence of the temperature of boiling and of the driers used on the properties of boiled linseed oil. The oil used was expressed in the cold from Calcutta seed; in colour it was a very pale yellow. In each experiment 50 c.c. of the oil, *i.e.* 45·7 grms., were heated in a glass vessel on the sand-bath, the sand reaching half-way up the layer of oil. The contents were stirred several times during boiling; the driers were previously dried. The best results were obtained when the temperature of the sand-bath was 230° to 275° C. After boiling, portions of the oil were spread on glass and allowed to dry in the open air in a good light. The coating was regarded as dry when it could be touched without retaining the impression of the finger. The results are given in the following table:—

Drier.	Quantity of Drier.	Duration of Boiling.		Temperature of Boiling.	Time of Drying.	Appearance of the Dried Layer.
	Grms.	Hrs.	Mins.	° C.	Hrs.	
Litharge	1·0	2	15	220	6	Almost colourless.
,,	0·2	2	15	250	10	,, ,,
,,	0·8	1	30	250	10	,, ,,
Lead peroxide	1·072	1	30	220	several days	Strongly coloured.
,, chloride.	1·247	2	30	250-360	24	Slightly ,,
Red lead	1·024	2	30	220-285	24	Strongly ,,
Lead oxalate	1·323	2	15	300	did not dry	,, ,,
,, tartrate	1·6	2	15	270	24	,, ,,
,, acetate	1·46	2	15	270	12	Slightly ,,
,, borate	1·105	1	30	220-300	20	,, ,,
,, carbonate	1·197	2	...	225	10	,, ,,
Zinc oxide	0·5	2	15	250	45	Almost colourless.
,, sulphate	1·987	2	30	285	45	,, ,,
,, sulphate	1·5	2	...	230	45	Yellow.
,, acetate	1·0	2	15	235-280	40	Colourless.
,, borate	1·0	2	...	240	40	Almost colourless.
,, borate	0·5	1	30	240	46	,, ,,
,, borate	0·5	1	30	240	46	,, ,,
,, nitrate	1·5	2	30	230	36	,, ,,
Manganese acetate	1·0	2	15	225-250	20	,, ,,
,, borate	1·625	2	15	220	20	Colourless and hard.
,, sulphate	1·72	2	...	240	40	Colourless.
,, oxalate	1·64	2	...	230	40	,,
,, acetate	0·5	2	...	225-250	20	Strongly coloured.
,, borate	0·5	1	...	230	20	Colourless.
,, acetate	0·5	1	15	225-230	20	,,
,, oxalate	1·5	2	30	230	36	,,
,, sulphate	1·5	2	30	240	36	,,
,, oxalate	1·0	2	45	240	48	Yellow.
,, nitrate	1·5	1	30	230	24	Blackish.
,, tartrate	1·0	2	30	230	24	Colourless.
,, formate	1·0	1	...	240	24	Slightly coloured.

Some of the boiled oils were examined in order to determine the percentage of metallic oxide they contained.

Drier.	Quantity of Drier.	Lead or Manganese per cent.		
		Calculated.	Found.	
			I.	II.
	Grms.			
Lead carbonate . .	1·197	2·030	1·392	1·477
,, acetate	1·466	2·230	1·396	1·338
Litharge	0·200	0·406	0·244	1·338
Lead borate	1·105	2·030	0·982	0·998
Manganese sulphate . . .	1·720	1·370	0·045	0·038
,, borate . . .	1·620	2·060	0·208	0·190
,, acetate . . .	0·500	0·347	0·248	0·256
,, oxalate . . .	1·500	1·034	0·248	0·030
,, tartrate . . .	1·500	0·670	0·049	0·043

From these experiments it might be concluded that oil boiled with lead compounds generally has a darker colour, which becomes more or less evident in the dried layer. Zinc compounds appear to exert no action on the oil; they make it dry slowly and impart no hardness to it. Manganese compounds give better results, but litharge the best; the oil dries rapidly, the coating is hard, and if the oil has not been boiled at too high a temperature it is barely coloured. Manganese borate and acetate are the best driers, especially the former; when the acetate is used, the temperature should not rise above 230° C., otherwise the oil becomes strongly coloured owing to the formation of tarry (?) products. The acetate is the best of the zinc compounds, yet the borate and citrate give good results. Chlorides, nitrates, and borates are not good driers, they cause too violent an action; the last-named decompose with difficulty, and require a high temperature. The use of formates, citrates, and tartrates appears to offer no advantage.

For the preparation of pale boiled linseed oil Rütgers proposes the use of glycerin. He states that manganous borate has the defect of possessing a low specific gravity, hence oil treated with it requires a long time to clear and deposit, and forms a considerable foot, which is to be regarded as loss. By utilising the solubility of manganous borate in glycerin these objections are apparently removed; the drying action of manganous borate is far greater when dissolved than when dry, thus considerably smaller quantities may be used to produce the same effect. The more anhydrous is the glycerin the greater is its solvent power. Nothing is said as to the method by which the linseed oil is treated with the glycerin solution, although this is very necessary, especially since glycerin does not dissolve in linseed oil, and could only be used as a means of transferring the drier. In order to produce a drying action, the affinity of the manganous borate for the linseed oil must be greater than for glycerin, in order that it may enter into the oil, which appears hardly credible.

Very interesting and suggestive are the results obtained by Livache with non-drying oils treated by his process. The drying and non-drying oils were treated with metallic lead and manganese nitrate, poured off, and then treated with litharge in order to remove excess of manganese nitrate. When these oils were exposed in thin layers, the drying oils were found to increase considerably in weight, but the non-drying oils very little. But when the oils were exposed for two years, it was observed that the drying oils, which had become solid elastic bodies, experienced a decrease in weight after one year; at the same time the coating, previously quite dry, became somewhat sticky on the surface. On the other hand, the non-drying oils, which only increased in weight after a long time, thickened considerably, and in certain cases formed a solid hard mass, which only slightly adhered to the finger. The alterations in weight and consistency are given :—

Oils.	Loss of Weight per cent. after		Remarks.
	One Year.	Two Years.	
Drying oils—			
Linseed oil . .	10·3	7·0	Somewhat sticky before drying.
Walnut oil . .	9·4	7·6	Rather ,, ,, ,,
Poppy oil . .	8·0	5·3	Flows readily ,, ,,
Cottonseed oil .	6·3	4·5	... ...
Beech-nut oil .	6·1	5·0	Adheres to the finger before drying.
Non-drying oils—			*Condition after two years.*
Colza oil . .	6·0	5·3	Solid, transparent, adheres to the finger.
Sesamé oil . .	5·2	4·8	Solid, transparent, adheres to the finger.
Arachis oil . .	5·7	5·6	Very thick, remains sticky.
Rapeseed oil. .	5·8	5·4	Solid, transparent, adheres to the finger.
Olive oil . .	5·3	5·7	Very thick, remains sticky.

THE MANUFACTURE OF BOILED OIL AND THE APPARATUS THEREFOR.

In the present condition of the manufacture of boiled oil, when there are the most varied views as to the necessity and desirability of using high temperatures, when numerous solid driers, liquid siccatives, and "siccative extracts" (generally solutions of linoleates or resinates of lead and manganese) are employed, when also the most different and apparently equally favourable results are obtained, it is difficult to take up and defend a one-sided position, especially since exhaustive information is expected in a treatise on the subject. The manufacture of boiled oil will therefore be treated in three sections, according to the temperatures employed:—

1. The preparation of boiled linseed oil at temperatures up to 30° to 40° C.

2. The preparation of boiled linseed oil at 130° to 140° C.

3. The preparation of boiled linseed oil at 220° to 270° C.

Halphen divides the processes used in the manufacture of boiled oil into five classes:—

1. The preparation by the action of heat alone, without any additions.

2. The preparation by means of manganese oxides, hydroxides, and salts. (The processes of Binks, Grohmann, Chevreul, Dullo, Barruel and Jean, the process with manganese resinate.)

3. The preparation with lead oxides or salts, with or without heating (with litharge, white lead, lead sulphate, Liebig's process, Binks' process (lead oleate), with litharge and zinc sulphate, with granulated lead and tin, with precipitated lead).

4. The preparation with lead, zinc, tin, or their salts.

5. The preparation by means of acids (hydrochloric and nitric).

Halphen recapitulates or abstracts, however, only those processes which have been more or less frequently mentioned in the literature of the subject, without regard to the fact that many of them are quite worthless. Such, for example, as the processes with granulated lead and tin, also no one to-day tries to make boiled oil simply by heating. The author does not say that the methods for the preparation of boiled oil by means of lead oxides and manganese compounds no longer have their application, but the processes as given by Halphen produce results extremely incomplete for practical purposes. Acids also are not used in the preparation of boiled oil; occasionally they are used for refining, though rarely. There is such danger of the decomposition of the oil without great care, when effective quantities are used, and the difficulties of entirely removing the acid from the oil are so considerable, also the loss occasioned by emulsified oil is so great, that this process is quite out of the question in practice.

The driers will first be considered. In the preceding section we have seen that the following substances are now in use for this purpose: litharge, red lead, lead resinate, lead-manganese resinate, lead oleate, lead-manganese oleate, crystallised and dehydrated lead acetate, pyrolusite, manganese peroxide, manganic hydroxide, manganous borate, manganese resinate, manganese oleate. All other driers are without technical application, though they may occasionally be used without producing particular results.

Pale boiled linseed oil can only be obtained with anhydrous lead acetate and manganous borate. All other driers produce more or less dark oils, especially when used at high temperatures. Boiled oil produced at ordinary temperatures or below 230° C. can only be used for the grinding and thinning of paints or for application alone. The varnish manufacture requires boiled oil, which has been heated at least to 270° C., so that all the foreign matters contained in the oil have been removed. A difference must therefore be made between boiled oil intended for painting and for varnish making.

It would always be advisable, speaking generally, to heat linseed oil up to 270° C. before converting it into boiled oil by some method or another. The foreign substances, which are always present, then separate, as has been repeatedly remarked; the intense yellow colour of the oil is changed to a pale green, so that it may be used in white paints; it acquires a somewhat burnt smell, which is liked by the consumers; it also deposits a remarkably small foot, especially when the conversion into boiled oil is accomplished without any considerable rise in temperature and liquid driers are used, whether they be true siccatives from linseed oil lead plaster, or solutions of lead resinate and oleate and manganese compounds in linseed oil or turpentine. The oil is heated in a simple manner, it is brought up to 130° C. in a pan of suitable size, maintained at this temperature until it contains no more moisture, *i.e.* until the oil no longer froths or foams, and only gives off small bubbles. A more rapid fire is then applied, so that the temperature rapidly rises to 270° C., and the decolorisation of the oil occurs, the colour changing from deep yellow to pale greenish. The heating of the oil is then discontinued, the fire is extinguished, and the oil allowed to cool. When quite cold it is drawn off, or run off from the deposit by means of a tap.

The quantity of the drier and its eventual combination are very important in boiling linseed oil. In regard to the colour of the finished oil, it must be said that it is useless to associate lead compounds with, *e.g.*, manganese borate, for the oil will not thereby be made paler. It is equally useless to the drying power of boiled oil to overload it with driers, for experience has shown that the excess of driers thickens the oil and forms foots, without increasing the drying power.

The permissible maximum quantities of driers are as follows:—

Litharge, very finely ground . .	2–3 per cent.
Red lead	2–3 ,,
Lead acetate, fused	2·5–3·5 ,,
Pyrolusite	5 ,,
Manganese peroxide	2–3 ,,

Manganic hydroxide	2–3 per cent.
Manganous borate	1–2 „
Manganese or lead linoleate . .	2–3 „
Manganese resinate, precipitated .	1–2·5 „
„ „ fused . . .	5 „

In the case of the last two driers, which possess very different powers of transferring oxygen, the origin indicates the necessary quantity.

The apparatus used in boiling linseed oil is a pan of sheet iron, cast iron (enamelled or not), or of tinned copper. In shape it differs greatly, as has been previously mentioned. It is advisable that it should have a greater width than depth, so that a large surface may be exposed to the action of the air. The size of the pan varies in accordance with the scope of the works, generally its capacity will be 50 to 500 kilos.; pans of a capacity of 1000 kilos. and more are, however, used. The smaller pans are generally arranged so that they can be readily removed from the fire, whilst the larger pans are built in and provided with special safety appliances to guard against fire and to prevent the heated oil from overflowing. For the former purpose a heavy iron cover, running on rollers and rails, is fastened to the roof of the room, and can be let down in case of fire, which it quickly extinguishes by excluding air. A run-off cock is also provided in the bottom of the pan, by which the oil can be rapidly drawn away to a cold tank. The arrangement for preventing the oil from running over, or rather, to prevent escaping oil from reaching the fireplace, consists of a circular channel running round the mouth of the pan, in which overflowing oil is caught, to be then carried away by a pipe. Two pans in juxtaposition can also be used; the oil is heated in the larger pan, which is of such a capacity that the heated oil fills it to within about 10 cms. of the edge. It is built so that oil which flows over cannot reach the fireplace, and so that it is surrounded by the fire and fire gases to only half its height. It is provided at the side with a pipe through which the oil, which would otherwise overflow, can run into the second pan, placed near and at a lower level, which is not heated. The pan may also be covered during the operation with a well-fitting lid, provided with a slide and elongating to the pipe which leads the vapours and gases to the chimney, where a small fire is maintained to burn the gases. The use of steam, instead of an ordinary coal fire, for heating the linseed oil has particular advantages, and if it is not yet employed to the extent it deserves, this is due to the cost of erection, which is fairly high owing to the necessity for a steam boiler. The oil is generally boiled in jacketed pans, more rarely in pans heated by a steam coil, with which it is difficult to attain the necessary temperature. The steam is either at an ordinary temperature or superheated; the use of the latter is associated with difficulties and dangers, but has increased with recent years, so that many oil boilers now use superheated steam. The apparatus heated by steam, and also other special constructions, will be described later in another place.

The preparation of boiled linseed oil at temperatures up to 30° to 40° C.—Large pans are not required for heating the oil; small pans, iron tanks, or even wooden tubs may be used. The treatment of the oil proceeds at the ordinary temperature of 10° to 12° C., but is much accelerated if it is possible to obtain a temperature of 15° to 20° C. by heating the room or by adding heated oil. The process is very slow at temperatures below 10° C., and since rapid production is generally desired, heating by one of the above methods is to be recommended. In this method of "boiling" oil only those substances can be used as driers which really dissolve in linseed oil—the resinates and oleates of lead and manganese and their solutions in linseed oil or turpentine, also liquid driers. The use of any other driers is quite impossible, since they exert no action on cold oil. The fused resinates and oleates of lead or manganese, or of both metals, are powdered as finely as possible and completely dissolved at 90° to 100° C. in a portion, about 10 per cent. of the whole, of the quantity of oil which is to be "boiled." The solution is then stirred into the remaining oil. If the driers are simply stirred in without being previously dissolved, turbid oil is produced, which brightens with difficulty, since the fused resinates and oleates do not dissolve in quite cold oil. On the other hand, precipitated resinates dissolve, and also the commercial solutions of resinates and oleates in linseed oil or in turpentine. These substances may be used without heating; they are added to the oil contained in a tank, the oil is well stirred repeatedly for 24 hours, and then left to settle. The time requisite to obtain a pure bright oil depends partly on the purity of the linseed oil, partly on the nature of the drier, and also on the temperature at which the oil is maintained whilst settling. Definite statements cannot be made; but if the mixture is sufficiently warmed and the drier of good quality, the oil brightens completely. The clear oil is then drawn off from the foot, which is collected in other vessels and allowed to remain at rest, so that more clear oil may separate.

The preparation of boiled linseed oil at temperatures between 130° and 140° C.—In this process the oil is heated in a pan to 130° to 140° C., and maintained at this temperature for 3 or 4 hours—a period sufficient to give a good product. All the driers may be used, but again the resinates and oleates are especially applicable; they are added to the oil either from the commencement of the operation, or after it has been heated for 3 or 4 hours at 130° to 140° C. In the former case darker and more viscid oils are obtained, in the latter the boiled oil is paler in colour and more fluid. The drying power is the same in both cases, and there is also no great difference in the hardening of the oil. When manganese borate is used, the temperature should never exceed 130° C., if pale boiled oil is required. In order to obtain a very pale oil, so-called "zinc-white boiled oil," the oil is heated to 270° C., and when cold separated from the deposit, heated to 130° C., and the manganese borate, ground in a little linseed oil, added with stirring. This process is sufficient to yield a pale boiled oil, which dries well.

The proportions of the driers to the linseed oil are as follows:—

Litharge, finely ground	2·5	per cent.
Red lead	2·5	„
Litharge, 1·5 per cent.; red lead, 1 per cent.	= 2·5	„
Litharge, 1 per cent.; dehydrated lead acetate, 2 per cent.	= 3·0	„
Pyrolusite	5·0	„
Manganous borate	1·5	„
Manganese resinate, precipitated	1·5	„
„ „ fused	3–5	„
Manganese or lead oleate	2·5–3	„

Liquid driers may also be used when the oil is heated to 130° C.; the maximum quantity should be 5 per cent., in order to obtain an oil drying within 24 hours. It should be remarked that all liquid driers obtained from lead plaster cause a considerable amount of foot, to which fact regard must be given.

There is no danger in working at 130° to 140° C.; it is only necessary in using pans heated by fire, not to fill them too full or heat too suddenly, so that the oil does not froth up—especially when it contains much water—and, flowing over the side of the pan, reach the fire. The oil in the pan can never take fire.

The preparation of boiled linseed oil at temperatures between 230° and 270° C.—With care and forethought this method of boiling oil also is without danger, since the boiling point of linseed oil is not attained, and the temperature of ignition (400° C., or above) lies far higher. The arrangements for heating have been mentioned previously. The quantity of driers is maintained within the permissible limits; they are introduced either when the oil is first heated or after it has been maintained at 270° C. for 3 to 4 hours, and has cooled down to 130° or 140° C. Not much remains to be said about this process; it is very similar to the immediately preceding. The boiled oil is generally dark and viscous, and possesses a somewhat unpleasant smell. In boiling linseed oil at high temperatures there is danger that men, who are continuously employed, may contract lead poisoning. Precautions should be taken, and the men changed at intervals.

The preparation of boiled oil by means of steam.—Either steam at a usual pressure or superheated may be used for boiling linseed oil. Several suitable forms of apparatus are described below. The process is very similar to the two previously described; the oil is heated for the same length of time, and a similar quantity of driers is used. The boiling process is without danger. The oil produced differs in quality from other boiled oils only in possessing generally a paler colour; it does not dry more slowly or more rapidly.

Jacketed pans.—Steam-jacketed pans, which are particularly suitable for preparing pale boiled oil, are made of boiler plate 4 to 5 mm. thick, capable of resisting a steam pressure of 4½ to 5 atmospheres. The steam is supplied from the boiler by means of a single steam pipe. Each pan has a safety valve, and one of the two is provided with an exit pipe for the steam, and with an outlet for the condensed water, both provided with cocks. Steam enters the apparatus first by *c′* into the pan B (Fig. 18), and thence proceeds into A.

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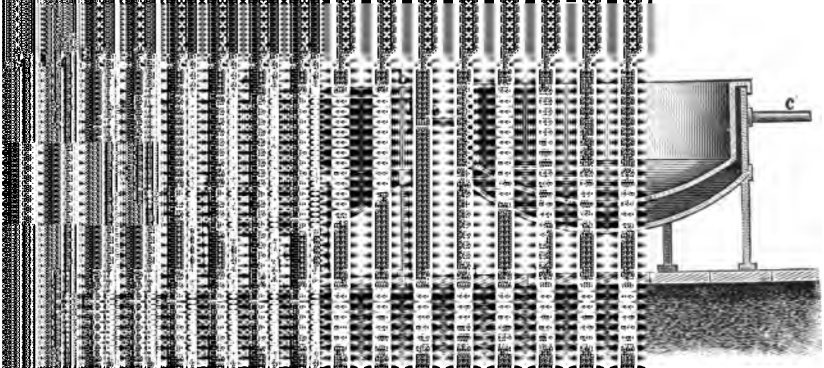

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**Apparatus for boil-
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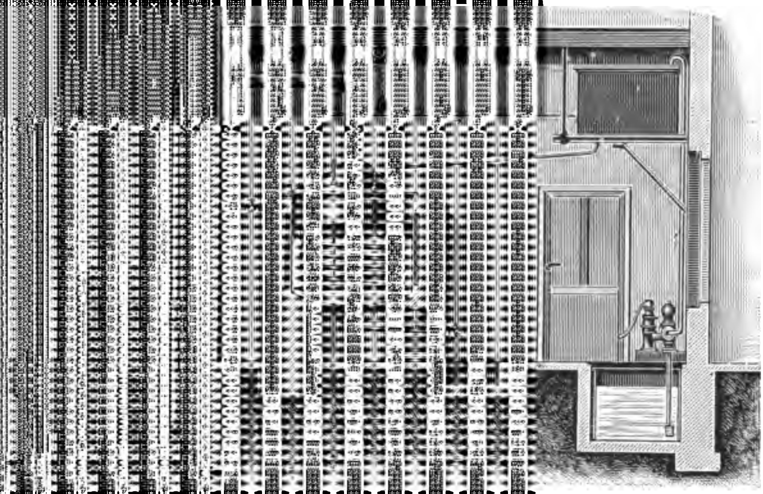

of Superheated Steam.

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are rapidly cooled

in their long course, are not inflammable when cold. A further protection against the flame striking back is obtained by placing fine wire gauze at the point where the vapours enter the ashpit under the bars of the superheater; the wire gauze performs the same function as in the miner's safety lamp. By this simple arrangement the process is made practically free from smell.

In boiling linseed oil by means of superheated steam there is practically no danger of the oil boiling over; but such an accident would entail material loss, therefore, in order to avoid every possibility, the pan is provided with an overflow pipe of sufficient width, which conveys the oil without any loss or damage to a tank at one side, where it cools.

In order to utilise to the greatest extent the heat of the steam, which leaves the coil of the boiling pan still hot, a tank is arranged at a higher level, in which the oil is heated for the next boiling. The process is thus made very economical, and the capacity of the apparatus is considerably increased, since the time is saved which would be required to heat up the oil in the pan from the ordinary temperature to that at which it leaves the preliminary heater. Another advantage of the preliminary heating of the oil, not to be despised, is that the pan is saved frequent great changes of temperature.

Apparatus for boiling linseed oil and removing the vapours.—The vapours may be drawn from the boiling pan by means of an arrangement consisting of a system of pipes with water tanks and jets (Körting's patent) introduced in the pipes at intervals. In the figure (21) the use of the jets is represented diagrammatically; *a* is the boiling pan, *b* the hood to catch the vapours. The system of pipes *c*, *d*, *f*, *g*, *h*, *i* communicates with the upper part of the hood. At the commencement of each of these pipes are jets *k*, *k*, . . . which are connected with a system of water pipes *l*, so that each has a branch pipe and tap. At the exits of the pipes *c* and *h*, or what is the same thing, the entrances to the pipes *d* and *i*, are the water tanks *m* and m^1, which collect the sprayed water and the precipitated oil particles. Beneath the tanks are the drainage pipes *n*, *n* . . . The system of pipes may be arranged in many different ways; it is proportioned to the requirements and to the number of jets *k*. To prevent the pipe *c* from burning, the hood *b* is surrounded by a water jacket *o*. At the entrance to the last pipe *i* a pipe *p*, with widened mouth, is placed so that the water projected from the jet k^5 may flow back into the tank m^1 without hindering the exit of the gases. The exhaustion of the gases above the pan *a* is proportioned to the needs of the moment, so that the vapours never escape the hood *b*; the room then remains free from noxious vapours and smell. The apparatus works as follows:—The vapours evolved from the pan *a* are exhausted by means of a draught created by the jet *k*; they are then intimately mixed with the spray produced by the jets, by which they are cooled and precipitated. The volatile vapours condense and collect in the tanks *m* and m^1.

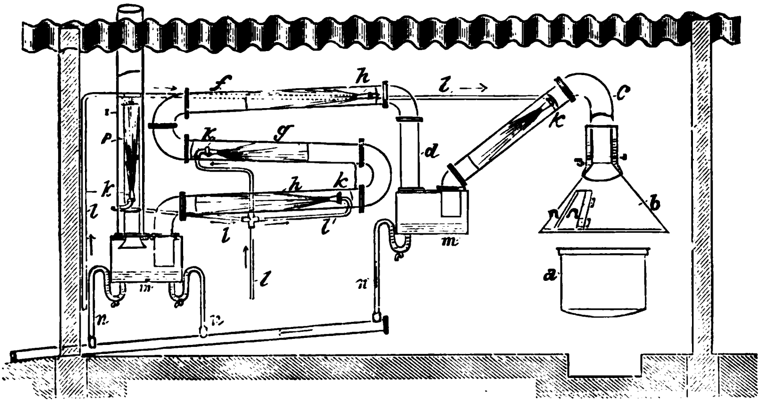

FIG. 21.—Apparatus for condensing the Vapours evolved in boiling Linseed Oil.

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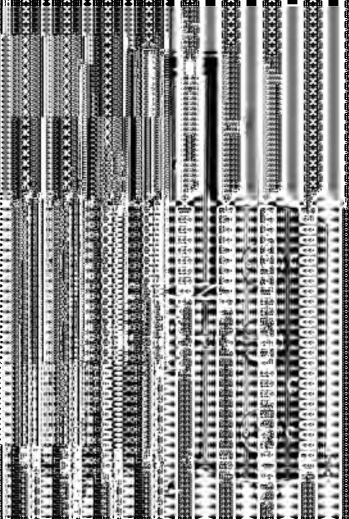

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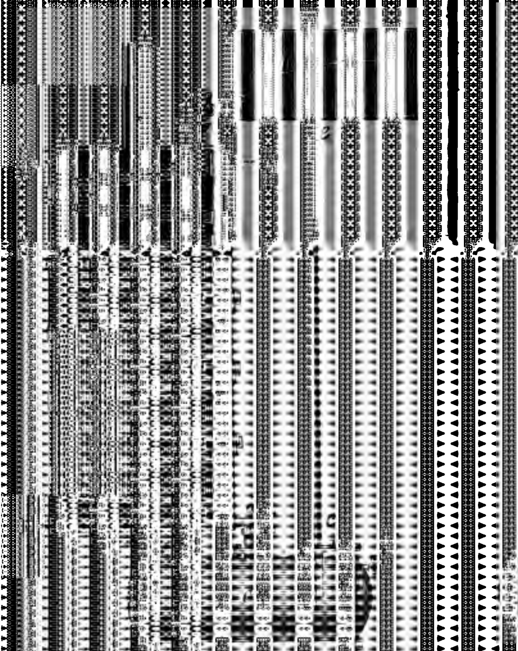

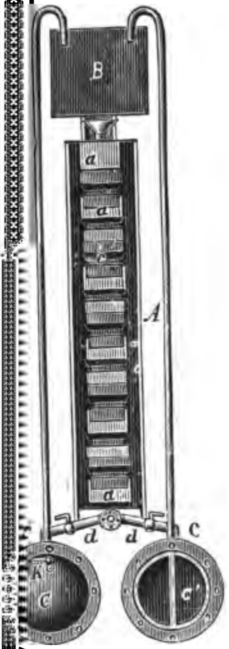

24.—Rapid boiling Apparatus. Zimmermann and Holzwich. Longitudinal Section.

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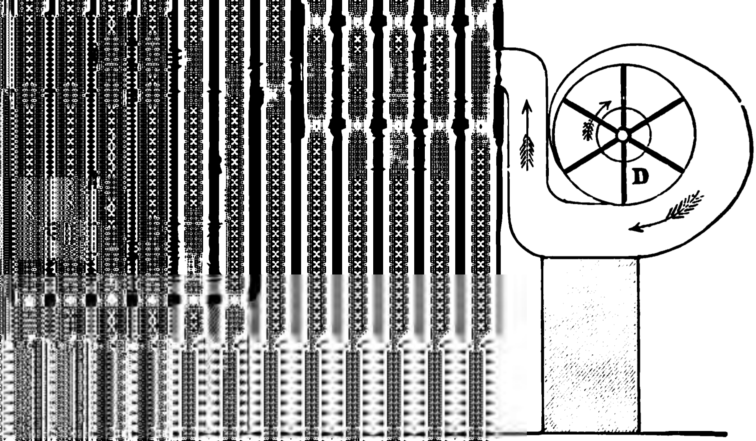

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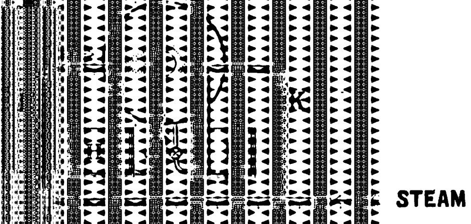

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[illegible] **onverting oil into** [illegible] e pan A (Fig. 25), [illegible] oil in the pan can [illegible] ans of the pipe R a current of heated air may be led into the oil; it is divided into small bubbles by means of the plate C, which is perforated by small holes. Hydrogen peroxide and a concentrated aqueous solution of zinc chloride are introduced into the oil as a fine spray by means of the current of air; the former is contained in the vessel H (Fig. 26), the latter in I. The pipe K introduces a current of steam or, better, of compressed air into the sprayer. The air forced into the oil from E by means of the pipe R thus carries both liquids in fine spray. Whatever portions of the spray are not carried away by the air current deposit on the bottom of the vessel E, and are drawn off from time to time by the taps S. A diaphragm is provided at the bottom of the vessel E, so that the two deposits may mix as little as possible. The operation is continued until 0·5 kilo. of hydrogen peroxide and 1·2 kilos. of zinc chloride solution have been used to 100 kilos. of oil. These substances are introduced into the oil in a very finely divided condition, and are very intimately mixed with it by means of the stirrer. The zinc chloride exerts a refining action, the hydrogen peroxide a bleaching action, and in addition the oxygen of the air an oxidising action, which converts the oil into "varnish."

LIVACHE'S PROCESS FOR PREPARING A GOOD DRYING OIL AND ITS PRACTICAL APPLICATION.—The process of Livache for treating linseed and other drying oils in the cold with unoxidised finely divided metallic lead, obtained by the action of metallic zinc on lead acetate solution, is based upon his researches on the absorption of oxygen by the drying oils.

The process and proportions for 100 kilos. of linseed oil are as follows:—3 kilos. of lead nitrate are dissolved in 15 litres of water, 6 to 7 c.c. of nitric acid are added, and then about 600 grms. of zinc in strips. The precipitated lead is brought into a large vessel containing bundles of wood fibre and rapidly washed with water, an operation which is several times repeated. The water is then poured off and a small quantity of linseed oil added, which is absorbed by the porous lead and displaces the water. When the water has been removed the mixture of lead and oil is poured into 100 kilos. of linseed oil. The mixture is continuously stirred; if a mechanical stirrer is not employed the oil and lead are brought into a barrel, which is violently rolled about. Immediately the oil comes in contact with the air and is oxidised, it acquires a red colour. If the operation has been properly performed the oil is as fluid as at the commencement; it contains a small quantity of lead, and dries in 24 hours. It is of advantage to introduce a manganese salt into the treated oil. For this purpose a very soluble manganese salt, manganese nitrate, is added to the oil, in the proportion of 1·5 kilos. to 100 kilos. of linseed oil; the oil is well shaken or stirred several times during 2 or 3 days, it is then allowed to settle and drawn off when clear. It contains an excess of lead nitrate, which indeed makes it dry but leaves it turbid; 750 grms. of precipitated lead are added and the oil well stirred. After standing and drawing off, the oil is quite bright; when exposed to the air in thin layers it dries in 6 hours. It should be observed that the oil acquires, in consequence of the presence of manganese, a brown coloration during drying, which soon disappears and then leaves the layer completely colourless. According to Livache, some manufacturers who employ this process have found in certain cases that the dry layers of oil were somewhat soft, which may perhaps be due to the presence of small quantities of glycerin. The drawback is removed by heating the oil to a moderate temperature after it has been treated.

Livache makes the following further remarks in regard to his process:—The study of the action of certain finely divided metals upon the drying oils has shown that the drying power is considerably increased when lead is used, and also in much smaller degree when copper is employed. The other metals precipitated from solutions of their salts have no appreciable action. On the large scale, oils with the maximum drying power are made by means of red lead or litharge; manganese is preferred in the form of borate. These facts have led to endeavours to introduce into the drying oils either the manganese compounds or the different metals, the action of which is to be investigated, without using them in a condition of the finest division. This object may be accomplished by treating a drying oil, made by the ordinary process or by means of finely divided lead, with the solution of a salt which produces an insoluble lead salt by double decomposition. For example, if linseed oil which has been treated with metallic lead, and thus contains that metal in solution, be treated with an aqueous solution of zinc sulphate, an oil results which no longer contains lead, but in which the presence of zinc can be clearly shown. Similarly, by using the sulphate of manganese, copper, etc., an oil is obtained in which the lead has been replaced by manganese, copper, etc. In this manner numerous experiments were made; the results obtained in regard to drying power are as follows:—

If an oil, which has been treated with lead, dries on glass in 24 hours, another portion of the same oil, in which the lead has been replaced by manganese, dries in 5 to 6 hours. If the lead be replaced by copper the oil dries in 30 to 36 hours; by nickel, iron, chromium, etc., in 48 hours. In treating the oil, under the conditions given, with solutions of salts, such as manganese sulphate, an emulsion is obtained from which the oil separates very slowly. Towards the end of the operation the oil must be gently heated to facilitate the separation. In order to avoid this inconvenience, Livache added powdered manganese sulphate to the oil treated with lead, and obtained an oil which, after treating for some time at the ordinary temperature, possessed extraordinary drying power and was obtained clear by a simple decantation. Thus the same result was obtained by the use of dry salts, but the oil contained not manganese alone, but lead also. How is this process to be explained? When raw linseed oil is treated with solid manganese sulphate no increase of drying power is obtained, although the oil has dissolved a certain quantity of the salt. But if an oil is taken which contains dissolved lead, a double decomposition occurs, the lead is replaced by manganese and an insoluble lead salt is precipitated. The process is similar to that which occurs on the large scale when manganese borate is allowed to act. Whatever process be used to introduce a manganese salt into a drying oil, the product dries in thin layers, as we have said, in 5 to 6 hours; if thick layers of dried oil be required, the operation requires months at the ordinary temperature. The oil may be thickened by destroying the skin as soon as it is formed, and thus allowing the air to have continual access, by which means the oxidation proceeds, but complete solidification is attained only with difficulty. When heat is applied the action proceeds more rapidly, but always requires a long time to reach the condition of complete solidity.

The practical value of Livache's process for producing boiled oil or a "good drying linseed oil," which makes it entirely suitable for manufacturing purposes, has not hitherto been sufficiently appreciated. The incomplete accounts of the process which have appeared in various periodicals do not seem to have borne fruit. Yet by this process it is possible to make linseed oil of good drying properties, which not only dries rapidly, but also always dries in a certain time —which depends upon the quantity of driers—when applied in layers of the same thickness, whether the atmosphere be moist or dry, or the temperature 10° or 35° C. This oil possesses, at the same time, another considerable advantage over oils prepared by boiling, or rather, by heating: it is merely linseed oil which has been made, by means of the additions, capable of absorbing oxygen and drying; it has not been chemically altered by heating to a higher or lower temperature with lead, manganese, or other compounds. For this reason it offers, when dried in thin layers, a far greater resistance to the destructive action of the atmosphere than true boiled oil. Paints prepared with this drying linseed oil, which has not been chemically altered, are far more durable.

The defects, which the products of Livache's process are supposed to possess, are their content of lead and a certain stickiness, which does not completely disappear after long drying. These defects have no real existence. In the first place, the quantity of lead actually introduced into the oil is very small, barely 0·25 per cent., since the greatest quantity added is only 1 per cent., of which at most only one quarter is dissolved, whilst the remainder sinks to the bottom. In the second place, the softness, which is peculiar to all unboiled oils, may be readily removed by heating to 270° C. before treatment, when the substances which prevent the oil from hardening are separated. It should also not be forgotten that the 0·25 per cent. of lead may be still further reduced by treatment with another metallic compound. The objections, which have been recently urged against the lead contained in boiled oil, are applicable only in those cases in which the linseed oil has been overloaded with lead, or in which lead compounds have been used, as, for example, crystallised lead acetate, which may injure the oil by reason of other compounds which they contain (acetic acid). Linseed oil which has been boiled with 3 to 5 per cent. of lead oxides, often at the much too high temperature of over

300° C., and which contains, in the form of lead oleate, the greater portion of the lead in those compounds, may be of short durability under certain conditions, but the durability of linseed oil cannot be increased or diminished by a quantity of lead which is 0·25 per cent. at the maximum. For these reasons alone, quite apart from the very simple method of preparation, the author recommends the introduction of Livache's method. If the linseed oil be heated to 270° C., as previously mentioned, allowed to cool, separated from the deposit, and then transformed by Livache's method into a "good drying oil," coatings of the oil, with or without a pigment, will dry *well and hard*, and will never be sticky.

Now, in regard to the practical application of this process, objections have also been raised, based upon the supposed complicated method for preparing the lead powder, which is not thought suitable for the large scale. One would, however, think that such a simple chemical process as the precipitation of a metal from a salt solution, washing and drying, would never offer serious difficulties. The whole operation is very simple, as we shall shortly see, and can be carried out by an ordinary labourer without special supervision.

The production of the lead powder is divided into the following operations: preparation of the lead acetate solution, introduction of the zinc strips, and removal of the deposited lead, washing, drying, and powdering. The lead acetate solution is prepared by stirring 10 parts of crystallised sugar of lead with 70 parts of water. When solution is complete, strips of clean sheet zinc about 10 cms. wide are hung in the solution, the end of each strip being bent into a hook, which is hung over the side of a vessel in which is the lead acetate solution. Immediately the zinc is dipped in the liquid it becomes black, and in a short time is covered with crystals of lead, which drop off or may be removed by hand when present in sufficient quantity. Thus fresh surfaces of metal are presented to the lead acetate solution, the sheet becomes gradually thinner, and finally quite disappears. Metallic lead and a solution of zinc acetate have been formed. When the zinc is dissolved, fresh strips of metal are hung in the liquid; they are renewed until all the lead has been separated from the solution, which is the case when a clean strip of metal dipped in the liquid does not become covered with the black coating. The lead lying at the bottom of the vessel is now collected, brought into a vessel with clean water, well washed, fresh water added, and the washing continued until no reaction for zinc acetate is perceptible. The water is then poured off, the lead well pressed in the hands and spread out to dry upon a flat surface, which is best exposed to the action of the sun. The lead may also be completely dried in a short time upon plates of gypsum or clay, which readily absorb water. The dried lead is now ground to the finest powder in a mill ("Excelsior Mill"); in this form it has the greatest activity, it should be kept in well closed vessels.

Lead is converted into the form of powder for electrical purposes by means of a jet of steam; this product may be used in Livache's process when the method of preparation given above appears too complicated, which it in no way is.

In preparing lead powder on a large scale by precipitation from lead acetate solution, the solution of zinc acetate obtained should not be run away, but should be concentrated, evaporated to crystallisation, and the salt sold. The cost of the process is then considerably reduced.

Now to consider the preparation of the drying oil itself. In the first place, it should be observed that Livache added the lead in the wet state to the oil; the author's numerous experiments have shown that this is not necessary, he endeavoured from the first to avoid the use of wet, that is, of freshly precipitated lead, well knowing the difficulty of brightening an oil which contains water in a state of fine division. The desired result is obtained with dry lead powder, but not with lead clippings or fine filings, which have no action upon linseed oil.

The largest quantity of lead powder which is added to the oil is 1 per cent., which is sufficient in conjunction with other oxygen compounds to impart to the oil the greatest attainable drying powder, that is, to make it dry hard in thin layers in 5 hours. This, or indeed any quantity of lead powder, has another action upon the oil in addition to imparting to it the capacity of absorbing oxygen from the air. In contact with the atmosphere, in thin layers of about 2 to 3 cms. deep, the oil is considerably thickened, and also bleached almost colourless in a very short time, often in a few days. This property of the lead powder does not seem to have been observed by Livache, at least there is no mention of it in his communications, yet it is very characteristic. It is evident in larger quantities of oil kept in loosely corked bottles, so that even the dark oils, treated with the largest quantities of lead powder and other metallic compounds, thicken appreciably in several weeks and become paler. There is no doubt that light plays a very considerable part in the change.

The author's numerous experiments as to the quantity of lead powder to be used have shown that larger quantities than 1 per cent. are quite useless, and do not further increase the drying power. This is again a clear proof that the limit of the greatest drying power of linseed oil is well defined, and that additions of substances, which transfer oxygen, beyond a certain point have no effect upon the drying power, and merely serve to overload the oil with metallic compounds, which may injure the quality and durability of dried coats of the oil.

If raw linseed oil, which has been tanked or heated to 270° C. and then separated from the deposit, be treated with 1 per cent. of lead powder for 24 hours with frequent stirring (in the case of small quantities by shaking in bottles) and then left to rest, the lead powder undissolved by the linseed oil in the given time separates together with certain impurities from the oil. The latter becomes pale and clear, and dries in thin layers upon glass or iron in 24 hours to a hard skin, which adheres well and is at the same time quite bleached, so that even a pure white paint would show no yellow tint. If linseed oil treated in this manner be ground with pigments, the paint would dry in 24 hours with an indifferent pigment; in 6 to 18 hours with lead pigments; and in 24 to 48 hours with those pigments which are known to retard the drying of boiled oil. Smaller quantities of lead powder than 1 per cent. produce an oil which dries in a correspondingly longer time; with less than 0·5 per cent. no difference can be observed from linseed oil which has not been treated with lead. Thus there is a minimum as well as a maximum for the quantity of lead powder.

If there be added to the oil, along with the lead powder, another metallic compound which is known to act as an oxygen transferrer, *e.g.* manganous borate, chemically or technically pure, or precipitated manganese resinate, the drying power is considerably increased, which is due to the manganese. Thus linseed oil treated with 0·75 per cent. of lead powder and 0·75 per cent. of manganese resinate gives a product, tolerably pale in colour, which dries quite hard in thin layers in 9 hours; with 1 per cent. of lead powder and 1 per cent. of precipitated manganese resinate the oil is at first comparatively pale, but darkens in a few days, its drying power is considerably increased; thin layers dry in 5 hours. More rapid drying cannot be obtained in this manner or by any method at present known. This is most clearly shown by the liquid driers, which are made by boiling linseed oil with 60 to 70 per cent. of lead oxides and diluting with turpentine, without which dilution a soft sticky mass, drying very slowly, would result.

The following products may be obtained by modifications of Livache's method:—

1. Linseed oil drying in 24 hours.
2. Very pale, almost white, linseed oil.
3. Very pale thickened linseed oil, reaching to the consistency of stand oil.
4. Linseed oil drying in 12 hours.
5. Linseed oil drying in 9 hours.
6. Linseed oil drying in 5 hours.
7. Thickened linseed oil drying in 5 hours.

The principle of the methods by which these 7 products are obtained is the same; the very pale and thickened oils require a further treatment, which is still to be mentioned. The author proceeds as follows:—Good tanked linseed oil, or linseed oil which has been heated to 270° C., cooled and separated from the deposit, is well mixed in the proportions, which will be given later, with the lead powder and the other driers, which have been stirred with a little linseed oil. An enamelled iron pan, a large tank or some similar vessel, is used, in which the oil is stirred at intervals of 1 hour during 24 hours by means of a stirrer driven by manual or mechanical means. It is then allowed to settle, so that it may become completely bright and clear, for which 4 to 10 days are required, according to the quantity; as a rule, 400 to 500 kilos. will brighten in a tank in 4 to 5 days. In this process the temperature of the oil, that is, of the room in which the process is conducted, is important. An experience of many years has shown that a temperature of 18° to 20° C. is the most suitable at which to effect the action of the driers upon the oil; higher temperatures do no harm, but have no appreciable advantage, whilst temperatures below 18° to 20° C. considerably hinder the transformation and make the brightening of the oil unnecessarily prolonged. At favourable times of the year a temperature of 18° to 20° C. will prevail in most rooms, in winter the room should be warmed by steam or fires, if rapid preparation of the oil is important.

The clearing process is considerably shortened if the oil is filtered, for which purpose the tube-filter, originally known as A. K. Stönner's, is suitable; it delivers large quantities of clear bright oil at a relatively small cost. In filtering that change is completed, which is peculiar to this process, namely, the darkening of the treated oil. All oils treated with the lead powder and other metallic compounds differ little, immediately after clearing, in colour from the original oil, but on standing, under the influence of air and sunlight, they become darker after a longer or shorter time; in the case of the oil drying in 5 hours, a product is obtained of a deep yellowish-brown colour, which is regarded by many consumers as a sign of "well boiled" linseed oil. This change proceeds in filtering, and at the same time the oil acquires the peculiar smell, which otherwise it would only obtain by long tanking, and which indicates to the experienced observer whether the oil has been boiled or prepared in the cold way. In the case of linseed oil which has been previously heated to 270° C., this smell does not develop, or not to the same extent. The smell is in no case such as to hinder the use of the oil, and is merely to be regarded as a characteristic sign of significance to the experienced.

1. **Linseed oil drying in 24 hours.**—

100 kilos. of tanked linseed oil, or oil heated to 270° C.
1 kilo. of lead powder.

If this oil, before it has settled, be placed together with the lead powder in shallow open vessels, in a layer of about

10 cms. deep, and exposed to the action of air and sun for about 14 days, there is obtained—

2. **Very pale, almost white, oil.**—This oil may be used for grinding the finest white paints: zinc white, white lead, or permanent white; quite white coats will be obtained. The bleaching process will continue without any further additions; in particular, the use of water is avoided, which causes much loss in bleaching oils. The oil may also be bleached by keeping in flat glass bottles, but a longer time is then required.

3. **Very pale linseed oil, thickened to the consistency of stand oil.**—The preceding oil, like all those prepared by Livache's process, thickens when exposed to the action of air and light. The same thickening occurs with ordinary boiled oil, and even with raw linseed oil, but it requires a very long time, and does not take place to the same degree.

In order to thicken the oil which dries in 24 hours, it is exposed in very shallow vessels in a layer 4 to 5 cms. deep for about 8 days, any skin which forms being removed, so that the air may have unrestricted access to the oil. The thickening is almost visible from day to day; when it has proceeded to the desired extent, the process is interrupted, the oil drawn off and preserved in tanks. To prevent dust from collecting, insects from falling in, and the oil from becoming dirty, the surface may be covered, at a distance of 4 to 5 cms., by a sheet of glass, or closely woven canvas may be stretched across. Air and light have then free access, and the oil remains clean. The thickened oil is very pale in colour, thick, and of good drying power.

The three varieties of oil next following are treated by Livache's process, not with lead powder alone, but with metallic compounds in addition. All the manganese compounds used as driers may be employed, the borate, acetate, carbonate, etc., zinc sulphate, etc. The author has obtained the best results with manganese resinate, which is used in the following processes:—

4. Linseed oil drying in 12 hours.—

100 kilos. of tanked linseed oil, or oil heated to 270° C.
0·5 kilo. of lead powder.
1·5 „ precipitated manganese resinate.

5. Linseed oil drying in 9 hours.—

100 kilos. of tanked linseed oil, or oil heated to 270° C.
0·75 kilo. of lead powder.
0·75 „ precipitated manganese resinate.

6. Linseed oil drying in 5 hours.—

100 kilos. of tanked linseed oil, or oil heated to 270° C.
1 kilo. of lead powder.
1 „ precipitated manganese resinate.

All the operations proceed in the same manner with all three oils, in the same time and at the same temperature. There is no difference in the colour of the oils immediately after they are bright. Under the action of light and air the colour changes, the oils become darker; the oil drying in 5 hours darkens most, the others less. This change of colour is not due to the action of the lead powder, but of the manganese resinate, for it occurs in the absence of the former substance.

The formation of a precipitate in the oil, generally known as a "foot," is extremely small, and frequently barely reaches 1 per cent.; it is rarely more than 2 per cent. Since the manganese resinate dissolves completely in the oil, we may assume with a fair degree of accuracy that the original quantity of raw oil is obtained as boiled oil or drying oil. But since the foot may be used together with the oil in grinding paints, there is really no loss in the process, which is to be regarded as the simplest and cheapest of all known methods for preparing boiled oil.

In the course of time, especially when air and light act without obstruction, the oil which dries in 5 hours undergoes a change; it becomes appreciably paler, finally even paler than the original raw oil, and becomes so thick when exposed to the air that it is converted into

7. **Pale thickened linseed oil drying in 5 hours.**—This oil is made in the same manner as the oil described under 3.

This, the author's, practical experience of the use of Livache's method in preparing boiled oil is first published in this place; the author hopes that it will be appreciated as its advantages deserve.

The preparation of "boiled" linseed oil by means of ozone and electricity.—In view of the increasing application of electricity in chemical industries, it is hardly surprising that it should be used for refining the drying oils and preparing boiled oil, for which purpose ozone is destined to play an important part. More than 15 years ago Schrader and Dumcke found by a number of experiments that it is only necessary to act with ozone upon raw linseed oil for a short time in order to bleach and thicken the oil simultaneously. The bleaching is completed when the ozonised oil is afterwards exposed for a day to the action of air and light. The "varnish" resulting from raw unbleached linseed oil is said to be water-white and to dry well; it is rapidly obtained in the cold without loss or danger. The gas is aspirated or forced through the oil in suitable vessels; any source of ozone which gives a good yield may be used. Later, Graf & Co. took out a patent for the manufacture of boiled oil by means of ozone; subsequently there have been several proposals to introduce ozone and the electric current into the manufacture of boiled oil, some of which will be given in detail. In ozonising, the current of ozone is simply led through the oil contained in a suitable vessel. When oil is treated by the electric current, it is emulsified with water or acidified water, and kept in continual movement. The two poles of the electric circuit are separately inserted in the liquid through which the current then passes. The firm of Siemens & Halske has repeatedly equipped such installations in Germany and Austria.

Process and apparatus for producing ozonised air on the large scale.—This process, due to E. Fahrig of London, for preparing ozone, or rather ozonised air, on the large scale, depends upon the fact that air, which is to be strongly ozonised, should (*a*) be quite pure, (*b*) contain much oxygen, (*c*) be absolutely dry, and (*d*) cooled. Air prepared in such a manner can only become completely ozonised when the silent discharge passes through it in a highly uniform manner.

According to researches and observations, a very constant electric current is required to produce ozonised air of regular and high efficiency. The methods and apparatus previously used for the preparation of ozone are in no way sufficient, since they do not fulfil these requirements; thus air ozonised in an absolutely uniform manner can never previously have been used on the large scale. The new process, which satisfies all requirements, is conducted in the following manner:—Atmospheric air is agitated by some suitable arrangement, and then led to a compressor, pump, Root's blower, or some similar apparatus, which takes in the air and forces it under high pressure into a system of pipes. The latter conduct the air first to a filtering apparatus, which is made to answer a double purpose: on the one hand, mechanical impurities, such as soot, dust, particles of coal, etc., are completely removed; on the other hand, by the separation of a portion of the valueless nitrogen, the air proceeding from the filtering apparatus is made considerably richer in oxygen. By decreasing the quantity and pressure of the air the quality is considerably raised; it becomes about 20 per cent. richer in oxygen than before. The purified and enriched air, which is still under sufficient pressure, then goes to a heating apparatus, where it is strongly heated, thence to a drying

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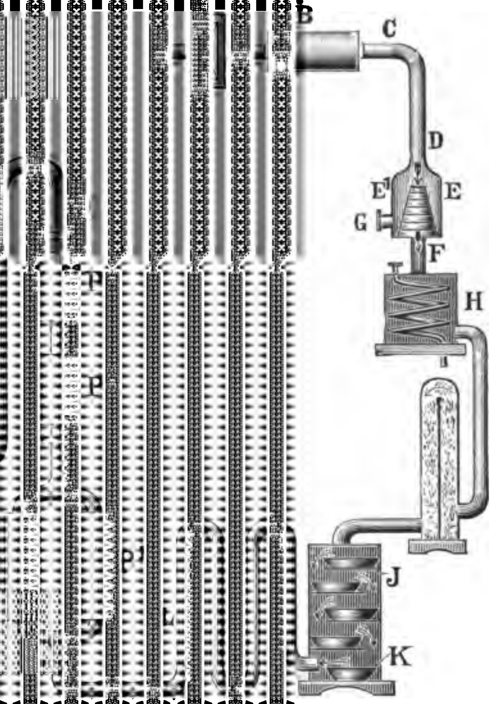

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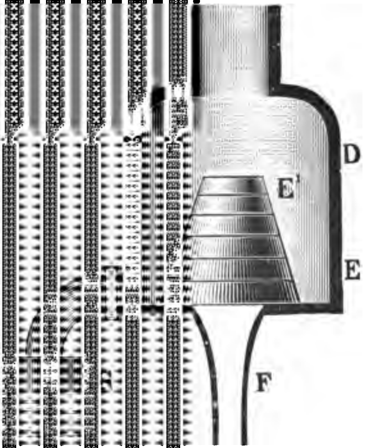

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and containing all the impurities escapes at G. The increase in the proportion of oxygen in the air is due to the difference in the rates of diffusion of gases, which depend upon the densities of the individual gases. Air very rich in oxygen is desirable in this process; the number and dimensions of the individual filter cloths must be arranged accordingly. The outlet G must possess a certain resistance, otherwise the whole of the air would escape and none be carried forward purified.

The purified air, rich in oxygen, is now led into the apparatus H, heated in the ordinary manner by means of a steam coil; the air may also be heated by other methods. The moisture in the heated air is then withdrawn by means of calcium chloride or pumice and sulphuric acid. The drying is completed in the chamber J, which contains dishes filled with strong sulphuric acid, arranged as shown in Fig. 27 so that the air always impinges upon the surface of the acid, flows along it, and then reaches the next dish, which it passes in the same manner. The air leaving this chamber, completely dry and still hot, is then cooled in a long system of pipes L to about 4° C., whence it passes to the ozonising apparatus P P^1 P^2 P^3, where it is subjected to the action of the silent discharge. The parts of the ozonising apparatus are joined together in such a manner that the air may pass through two or more at the same time, and thence into the last two parts, which it traverses in succession. The number of similar parts arranged in parallel may be varied, but after these not more than two should be placed, since experience has shown that further ozonisation does not occur, but rather that the ozonised air loses activity.

The difficulty, previously mentioned, of providing the high tension induction current necessary for ozonisation, is overcome by employing an alternating current dynamo M, excited by the constant current dynamo N. The former supplies a uniform current to the transformer O, which by its peculiar construction, especially in regard to the use of masses of iron, can neither become heated nor produce a current of higher tension than is necessary for the purpose.

The current produced in this way is utilised in the ozonising apparatus P P^1 P^2 P^3, shown in Fig. 29, in which P and P^1 are in parallel, and thus simultaneously traversed by the air, whilst P^2 and P^3 are in series, one after the other, and thus cause to a certain extent compression of the air passing through them.

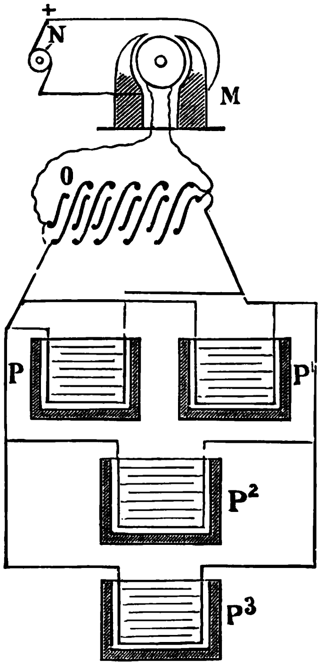

FIG. 29.—Apparatus for the Production of Ozonised Air. Ozonising Apparatus.

The single parts of the ozonising apparatus (Fig. 30) are so constructed that the silent discharge acts from the greatest possible surface and in the most uniform manner. The luminosity in the apparatus, which can be observed through spy holes, has different shades, according to the stage of the ozonisation. Thus the process can be followed and controlled by altering the current of air or the strength of the charge. The ozonised air passes through the pipe Q into the gas holder R, in which it is collected over water covered by a layer of oil.

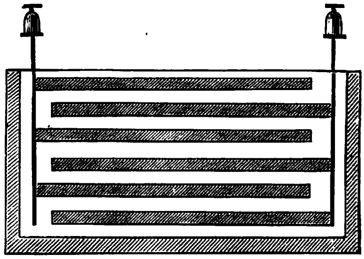

FIG. 30.—Apparatus for the Production of Ozonised Air. Ozoniser.

The advantage of this process consists, in the first place, in the production of air extremely suitable for ozonisation, characterised by complete purity and dryness and a high proportion of oxygen. Further, the preparation of ozone by this process does not necessitate the use of oxygen, but air, even if impure, may be used. The problem of the continuous production of a constant induction current of very high tension has been solved in a remarkably simple and successful manner.

Process of Müthel and Lütke.—In this process the drying oils are treated with gases, or mixtures of gases, which have been previously subjected to the action of electricity—highly oxidised and easily decomposable oxygen compounds, which at a low temperature are decomposed into nascent oxygen and lower oxides. The oxygen then oxidises the fatty acid compounds with which it is in contact. The following gases may be used in this process: a mixture of equivalent volumes of chlorine and steam, sulphur dioxide with air or oxygen in excess or in equivalent quantities, sulphur dioxide and nitrous acid, nitrogen and oxygen with steam, nitrous oxide with air or oxygen.

Some one of these mixtures of gases is subjected in the apparatus represented in the figure to the continued action of a powerful, dark electric discharge, by which the oxidation is carried to the highest point, if a sufficient quantity of the oxygen compound be present. A definite formula cannot be given for the product, since its chemical composition varies within wide limits with the quantities of the gases acting upon one another. For example, Cl_2 reacts with H_2O producing $2HCl + O$; in the action of O upon SO_2 the two gases must be present in such relative volumes that S_2O_7 may be formed.

It appears to be advantageous, in order to produce the highest degree of oxidation, to allow the oxygen compound to act in excess upon the oxidisable gas. The apparatus used

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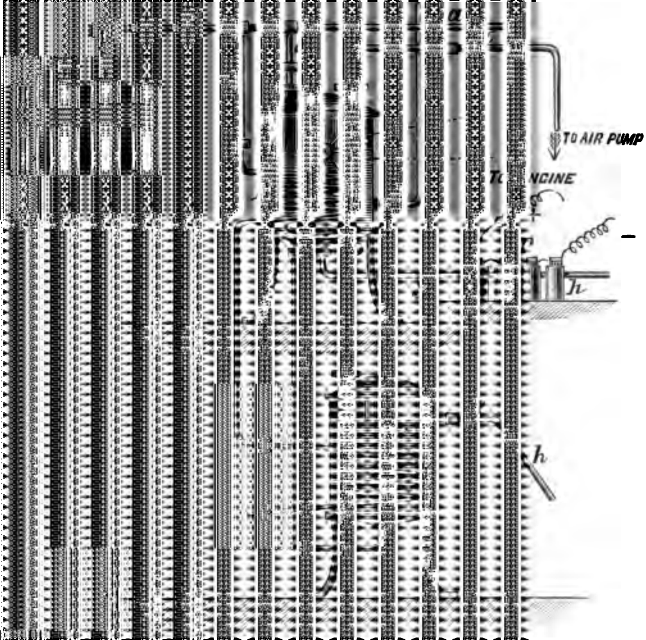

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diminished pressure. The paddle wheel C, driven by the engine, brings oil and gas into intimate admixture. By this means the decomposition of the fatty acid compound is remarkably accelerated, and in a relatively short time a pale fluid oil is obtained, which readily dries in the air to an elastic and solid mass. With the same success is used a mixture of nitrous oxide with air in the proportion of 2 to 1½ by volume, or even nitrous oxide alone is employed.

The use of the other gaseous mixtures already mentioned is not excluded; the object is always to produce, by means of electric discharges, nascent oxygen or highly oxidised oxygen compounds, which latter are decomposed only when they come in contact with the hot oil.

The products of decomposition, mixed with a small portion of unused gas, drawn away by the air pump, may either be regenerated or simply led under the fire-bars of the boiler. When the oxidation is finished, which is recognised from test portions, the introduction of gas is discontinued, the stirrer stopped, and after some time *d* is closed, whilst *e* is opened, steam then enters, which first fills the vacuum, and then, *f* being opened, drives the oil into the apparatus W, half filled with slightly ammoniacal water, heated by the steam which passes through the coil S′ after leaving S. The oil rises through the liquid and is freed from adhering acid, it is then forced through *h* directly into the storage tanks, if it is not thought advisable to cool it previously.

X **Storage of boiled oil.**—All boiled linseed oil, by whatever process prepared, is more or less turbid, owing to the constituents of the oil (mucilage, albumin, etc.) separated by the driers and also to the insoluble portions of the driers themselves. In the case of litharge, red lead, undissolved lead oxide, and metallic lead remain; in the case of manganese compounds, lower oxides of this metal; in the case of resinates and oleates, undissolved portions of these substances.

A turbid oil is not attractive to the buyer. For immediate use the turbidity is of little account, since the foot is also ground into paint. In order to obtain a saleable product, boiled oil must be cleared, which is generally done by storing in barrels or, better, in large uncovered iron or wooden tanks. The finished oil is brought as quickly as possible into the storage vessels, the oils prepared by heating as hot as is permissible, and there left at rest. The oil brightens according to the quantity and to the temperature in the storeroom in 8 to 14 days, the impurities settle to the bottom, and the pure oil is either drawn away or run off by means of a tap at a proper height in the tank, and brought into vessels for storage or delivery. Upon the bottom of the tank is found the foot, more or less viscous, and often still containing oil. This deposit is collected in vessels for the purpose, and left at rest; after some time clear oil rises to the surface, whilst the foot becomes more and more compact, and no longer gives up fluid. This by-product can be used in paint grinding and preparing cements, and also in the manufacture of soap. By means of powerful presses all the remaining oil in the foot may be pressed out and the residue used as fuel, for which it is very suitable.

All boiled oil, with whatever drier prepared, suffers change on long storing. The alteration is not always the same; with the same oil different results are obtained, according to the temperature and the varying action of air and of light.

All oils obtained by heating produce a deposit on long keeping, although they may be quite pure, quite clear and bright at first. The quantity of this deposit varies with the nature of the oil and of the driers. Oil boiled with lead gives, as a rule, more deposit, manganese oil less, oil prepared with manganese resinate the least, and in the latter case the oil prepared in the cold gives still less. This formation of deposit may take place to such an extent that more than a quarter of the oil becomes thick and turbid, without any clear explanation of the cause being possible. According to Weger, the formation of a deposit is connected with the decrease in drying power; the manganese boiled oils prepared by him were all bright, and had formed no deposit, or merely a trace. Of the oils boiled with lead and manganese compounds, those kept in closed vessels were bright, those in open vessels were turbid; the oils which had been heated showed almost no deposit (this is contrary to the author's experience). Oils prepared in the cold give less deposit when kept in open vessels than in closed. Weger deduces the connection between formation of deposit and decrease of drying power from the fact that manganese oils do not appreciably diminish in drying power and form no deposit, whilst lead oils, which form a foot, dry less rapidly in the course of time. The heated lead-manganese oils form less deposit than those prepared in the cold, and also decrease less in drying power. There are, however, other influences to be considered, in particular the result of excessive oxidation by the air, since of the heated lead-manganese oils, which always form little deposit, those kept in open vessels decrease more in drying power than those protected from the air; and of the oils prepared in the cold, those which form more deposit decrease less in drying power than those forming a small deposit. From this Weger concludes that a more stable incorporation is obtained by gentle heating than by working at the ordinary temperature; in the first case the amount of deposit is smaller and the drying power diminishes less than in the second. This inconvenience can under no circumstances be avoided completely with lead-manganese boiled oils, they always decrease in drying power, whether made cold or hot, pale or dark, and kept in open or closed vessels.

There is, however, less decrease when stored in closed than in open vessels, and less with oils prepared by heating than those made in the cold. The formation of a deposit in boiled oil during storage is a great inconvenience; in order to avoid delay in sale, it should not be kept in barrels upon the ground, they should be placed upon stands, and the oil drawn off into clean vessels for despatch.

Boiled oil also undergoes change in colour and consistency during storage. According to Weger's observations, there is no difference between boiled oil kept in the light and in the dark. The author regards this statement as correct only in so far as it relates to a short time and to considerable quantities, such as might be kept in large round glass bottles; in thin layers, as in flat bottles with a layer of oil 2 to 3 cms. thick, and on long standing, all boiled oil is bleached, whether made with lead or manganese. The oil made by means of lead powder is bleached in a short time, even in thick layers, although it may contain only traces of the metal. This is a peculiar action of lead which appears to have escaped the remark of scientific observers up to the present. The effect upon the bleaching of boiled oil kept in open vessels is under all circumstances very considerable, whether in the light or in the dark, and in this case again the oils prepared in the cold with lead powder and manganese resinate may be so far decolorised on very long standing that the oil becomes almost colourless. Simultaneously with the bleaching, a thickening of all boiled oils occurs, and this is the more considerable the greater the surface exposed to the air. According to Weger, heat has no effect here, but will naturally assist the change. He regards the alteration as entirely due to the action of oxygen and not of light also; all boiled oils kept in closed vessels preserved their fluidity. On the other hand, the lead-manganese oils stored in open vessels for a long time thickened very considerably; only two of the manganese oils, which were kept for a short time, were observed to thicken. Frequently an intentional admittance of air to boiled oil is unnecessary to produce thickening, the air in a half-full bottle is often sufficient to thicken the oil considerably. A skin forms under which the thickening proceeds, so that finally quite a viscous product is obtained. All these observations concern boiled oil kept for a long time, months, not weeks; after a lengthy period the alteration is perceptible to the eye.

The smell of boiled oil also undergoes a change; on long storage it becomes at once rancid and ethereal. The causes of the change and the reactions which occur have not yet been further investigated. The alteration in odour is particularly evident in a short time in the case of the oils treated with manganese resinate and lead powder in the cold. All such oils smell distinctly of raw linseed oil immediately and for many days after they are made, and even for months if they are kept in closed vessels, but if they are exposed to the action of the air, or filtered through filter paper or a tube-filter, the odour immediately alters. The smell is now so peculiar and characteristic that it serves to distinguish such oil, made in the cold, from that made by heating. The oil darkens simultaneously with the change in smell; the colour, which was originally, after preparation, and even after standing 8 to 10 days, but little darker than that of raw linseed oil, at once deepens on filtering and exposure to air, and the oil becomes as dark as an oil boiled for a long time with litharge.

The "boiled" oils prepared with liquid driers, simply by mixing the two fluids, change in a shorter time, and more considerably than the boiled oils made by means of solid driers. Whether the liquid driers consist of solutions of linseed oil lead plaster in turpentine, or of resinates of lead and manganese in linseed oil or turpentine, makes little difference; both solutions produce in linseed oil, on long standing, a fairly considerable quantity of deposit of whitish or yellowish colour, often gritty and friable, whilst the deposit formed in other boiled oils consists of fine soft particles. Frequently boiled oil made with liquid driers separates completely into two layers, a solid portion at the bottom of the vessel and an upper liquid portion, pure and bright.

In the oil made by the modification of Livache's method, after brightening, completion of the process, and exposure to the action of light and air, there occurs no further formation of foot or separation of driers (lead or manganese compounds). The oils, however, thicken and bleach in such a manner that, since the short duration of the process does not permit the separation of solid particles, only a pale oil remains, thick and of good drying power, and frequently somewhat opalescent.

Properties of boiled oil.—The properties which the consumer requires in boiled oil intended for use in paint and for coating wood, masonry, and metal (generally iron) are comprised in—(1) Consistency; (2) Colour; (3) Smell; (4) Taste; (5) Clearness; (6) Drying power; (7) Purity, that is, freedom from foreign substances—rosin, rosin oil, mineral oil, boiled oil substitutes, and other vegetable oils. In the following pages these characteristics will be described as briefly and precisely as possible. Although few users of boiled oil would test it by taste, yet this property affords an excellent method for recognising quickly certain impurities—rosin oil, mineral and fish oils, so that it will not be omitted.

(1) *Consistency.*—Good boiled oil, in whatever manner prepared, should be but little more viscous than raw linseed oil, otherwise it would not be suitable for forming thin coats of paint, and would have to be diluted with turpentine. In former years consumers laid great importance upon the thickness of boiled oil, which they regarded as a guarantee of a good drying and well boiled oil; more recent experience has shown that such thick oils have experienced a more or less deep-

seated decomposition, since they have been heated for too long a time and at too high a temperature. Such oils never produce quite hard coats, are applied with difficulty, and must be diluted with turpentine. All boiled oils made by Livache's method, and at temperatures not above 150° C., are thin; those which have been boiled for a long time, and at a high temperature, are, on the contrary, thick.

(2) *Colour.*—The colour of boiled oil depends upon the method of preparation, the temperature used, and the nature of the driers; it varies from the pale yellow of the palest boiled oil, through red to reddish yellow and, finally, reddish brown, but should never be dark brown or black, which last colours denote the use of too high a temperature. Oil boiled by means of steam is paler than that made over the fire. In general, oils boiled with manganese driers are of pale colour, whilst litharge, red lead and lead acetate produce dark coloured oils. Blown or ozonised linseed oil has generally a very pale colour. The duration and temperature of heating in the preparation of the oil have considerable influence upon the colour; all oils heated with the driers for a long time turn out dark. Boiled oil made by heating raw linseed oil to 150° C. as a maximum, and adding the driers as the temperature sinks, is always pale in colour. Frequently consumers prefer dark boiled oil, believing that oil "boiled well" and for some length of time with the driers must be better and dry more rapidly than pale oil. In preparing white paints and pale tints the colour of the boiled oil is important, since white paint is required to be pure white, and boiled oil gives it a yellowish or even brownish tint, which, however, is of no importance to the colour of the dried paint, since all boiled oil bleaches in drying, if light and air have sufficient access. The coating remains yellow in the dark; but even very pale boiled oil, which contains much lead, turns yellow in conjunction with white pigments, if light is

excluded; lead boiled oils should therefore not be used for white paint.

(3) *Smell.*—The odour of good boiled oil recalls that of raw linseed oil, but is at the same time empyreumatic, in this respect resembling the vapours evolved when the oil is heated. The odour of boiled oil made in the cold is peculiarly aromatic and ethereal, and an expert will at once be able to decide as to the method of preparation by means of this characteristic alone. The smell may be unpleasant, but should not be overpowering, still less may it have a similarity with the smell of rosin or fish oils, which are used as adulterants and can be recognised with certainty by other methods. If the oil has been so strongly heated as to cause partial decomposition it has a burnt and unpleasant smell, and a dirty brownish-black colour.

(4) *Taste.*—The taste resembles that of raw linseed oil, but is more biting, bitter, and unpleasant; adulteration with rosin or fish oils is most rapidly and easily recognised by means of the taste.

(5) *Clearness.*—All boiled oil, whatever its colour, must be quite clear, and in thin layers transparent; it should contain neither turbidity nor solid particles. Turbidity is generally due to insufficient storing; if oil is not clear it should be left at rest in a moderately warm place for at least 14 days, when it will brighten if it is not adulterated with rosin oil; from the quantity of the foot it may be decided whether the oil had been previously sufficiently tanked or not. Every boiled oil, however bright, gives a further deposit on long standing, the quantity of which should not exceed 0·25 per cent. after months, an amount which need not be considered. Frequently boiled oil is sent away a few days after it is made, and when it has only partially brightened, it will then reach its destination turbid; it will clear after standing 2 to 4 weeks, but the quantity of foot may reach

7 per cent.; such an oil is to be regarded at least as unsatisfactory.

(6) *Drying power.*—The drying power is one of the principal characteristics of good boiled oil. In an earlier chapter the oxygen absorption of boiled oil and the drying process have been treated in detail; the remarks in this place are restricted to general requirements in this respect.

Every boiled oil must dry, when spread upon glass or metal, in 24 hours at the longest, to such an extent that the finger slightly adheres, but does not stick. Boiled oil is made to dry in 5 to 6 hours, in 12 hours, and in 18 hours, but should never require more than 24 hours; when longer time is necessary the oil cannot be regarded as satisfactory. If pigments be mixed with the oil and the paint applied in a thin layer, the drying may be accelerated, as by white lead and red lead; may remain normal, as with indifferent pigments; or may be diminished, as by soot blacks, etc. Boiled oil is best tested by spreading it out in a thin layer upon a sheet of glass or of polished metal. The layer must dry as above given in 24 hours; it should be no longer sticky, but it should not be required to be hard, for the hardening of unmixed boiled oil is simultaneous with incipient decay. On the other hand, boiled oil must become hard when used with pigments, and should not be soft, presupposing that the paint has been made in the proper proportions. Boiled oil made in the cold, whether with liquid driers or with soluble driers, produces a somewhat softer coating than when made by heating, but gives a more durable coating of paint, because there has been no decomposition of the linseed oil, such as takes place by heating at high temperatures. In former times, when different views were held in regard to the formation of boiled oil and the drying process, products made simply by mixing with liquid driers were not regarded as boiled oil, for "in the preparation of boiled oil higher temperature and the introduction of oxygen, whether by the action of air or the reduction of added metallic oxides rich in oxygen, are absolutely necessary." This opinion is now obsolete; any "linseed oil made active," by whatever process, is regarded as "boiled" oil.

(7) *Purity.*—(Freedom from foreign substances—rosin oil, rosin, mineral oil, and boiled oil substitutes). Of every boiled oil brought into the market purity should be expected, if the price does not *à priori* show that it must be adulterated. As adulterants are much used: rosin oil, rosin, mineral oils (especially debloomed), and the different boiled oil substitutes, which are generally made upon the basis of rosin or rosin oil. Recently blown (oxidised) mineral oil mixed with petroleum, or petroleum ether, has been brought into the market as a boiled oil substitute. All these adulterants, as is shown in detail in another place, considerably affect the drying of linseed oil, and naturally also its durability. Boiled oil mixed with rosin and rosin oil, and even with small quantities of mineral oil, indeed dries, but remains soft, becomes sticky by the heat of the hand, and soon perishes on account of its softness. Boiled oil containing large quantities of mineral oil dries fairly normally, but the mineral oil separates on the surface; boiled oil containing fish oil does not dry at all. All these additions are objectionable.

Hurst gives the following methods for ascertaining the purity of boiled oil:—Boiled oil has a specific gravity of 0·941 at 60° F.; its flash point lies above 500° F. The flash point is determined in the same manner as given for raw linseed oil (p. 66); at times boiled oil is adulterated with the heavy mineral oils, which have a high flash point; these are recognised as follows:—10 grms. of oil are brought into a beaker with a little water and caustic soda solution, and the mixture heated. The linseed oil is saponified, whilst the mineral oil remains unaltered; a large quantity of warm water is added, and the whole transferred to a separating funnel, petroleum ether is added, and after shaking the mixture is allowed to settle. Two layers form, the lower an aqueous solution of the linseed oil soap, the upper a solution of the mineral oil in petroleum ether. The lower layer is drawn off, warm water again added, the funnel shaken, and the mixture allowed to separate. After drawing off the lower layer a further quantity of warm water is then added, the mixture shaken, and again allowed to separate. After drawing off the watery layer this washing is repeated until the ether layer is quite clear; it is then brought into a weighing glass, the ether evaporated on the water bath, and the residual oil, which forms the adulterant, weighed.

In practice, on the part of the consumer, it is frequently required that boiled oil shall dry in a certain number of hours; and in the same way, on the part of the manufacturer, it is definitely maintained that the oil dries in a certain time. The former demand is unfair, the latter statement is of no account, since in both cases a definite place and similar conditions of atmosphere, moisture, air currents, temperature, etc., must be presupposed. A boiled oil which dries on one day and in one place in, say, 10 hours, may require only 8 hours on another day in different weather, and again may require 12 to 14 hours on a third day. A boiled oil which requires 7 hours in one place may require more or less time on the same day in another place. In addition, there is another point to be considered in regard to the drying of boiled oil, or of the paints made from it. Boiled oil is rarely used alone, the pigments with which it is mixed influence the drying; thus all lead pigments, especially white lead and red lead, in less degree chrome yellow and chrome red, considerably assist the drying; whilst zinc white, the earth pigments to a greater extent, and the soot blacks to the greatest, retard the drying. Thus, in spite of using the same boiled oil, paints may require quite different times to dry, depending upon the chemical nature of the pigment. Consequently, no practical purpose is served when a consumer requires that boiled oil shall dry in x hours, or when a manufacturer puts his oil upon the market as drying in y hours. In each case a fairly considerable margin must be given to allow for the above-mentioned influences of atmosphere, moisture, temperature, and pigment. A definite statement is also not permissible, because the principal factors, light, temperature, and moisture, cannot be regulated at will. In order to obtain an accurate comparison of the drying of boiled oils, the different samples must be exposed, *in layers similar in every respect, at the same time and near one another*, to exactly the same conditions of air, moisture, light, and heat. Accurate results can then be obtained; the comparison of layers drying at different times and under other conditions is quite useless.

In order to obtain accurate results with paints, the same pigment must be mixed in the same proportion with the oils under examination, and *the layers must be as similar as possible and exposed at the same time near one another.*

In regard to statements as to the time of drying of boiled oil, Weger remarks: These figures cannot be regarded as absolutely definite, but must either be taken as liable to vary within wide limits or must be expressed as a ratio in comparison with a standard oil. This standard is obtained in the most simple manner by preparing it afresh each time with a definite quantity of driers, or a boiled oil is chosen which is certain to form no further deposit and not to decrease in drying power. Manganese resinate is the most suitable drier for this purpose. This substance is dissolved in 2 to 4 times the quantity of linseed oil, the solution filtered and finally made up to a certain definite percentage of manganese, say 1 per cent. of Mn. Of this solution a certain volume, say 10 per cent., is added to a *normal* linseed oil—the most difficult condition.

CHAPTER V

THE PREPARATION OF VARNISHES FOR LETTERPRESS, LITHOGRAPHIC, AND COPPER-PLATE PRINTING; FOR OILCLOTH AND WATERPROOF FABRICS

IN the manufacture of the various printing inks, of oilcloth, leather cloth and similar substances, of waterproof materials, and of linoleum, linseed oil is practically the only drying oil employed. Walnut oil is used in very small quantities, and perhaps wood oil alone has the prospect of finding extensive use in the place of or together with linseed oil. In the manufacture of all the products mentioned a substance differing essentially from ordinary boiled oil is required. The linseed oil or varnish employed must be syrupy and viscid. In the case of varnish for printers' purposes this condition is requisite, since a thin ink, or an ink ground with thin varnish, could not be printed, it would lack the necessary adherence—would be "short," and run off; in addition, thin varnish, when brought on to paper or other materials upon which it is occasionally used, produces grease marks, and the print would thus be unreadable. The varnish intended for printers' use, whether for letterpress, lithographic, copper or zinc-plate printing, for black or coloured inks, should also contain no driers. The inks are applied in such an extremely thin layer that they dry at once, being assisted by the absorbent nature of the paper, do not spread, and leave no oily edge round the printed letters. This rapid drying takes place in spite of the long time which the same ink would require to dry, if applied in layers of a thickness equal to that of an ordinary coat of paint. If driers were added in any form to varnishes intended for printing, there would be danger that the paper would adhere to the type and tear. The essential properties of a printing ink are given below; they are based principally upon the nature of the varnish, if we neglect the pigment and the properties it implies.

1. The ink must be a completely uniform syrupy liquid (for letterpress or lithographic work). It should contain neither skin nor other impurities which might be introduced by the varnish, nor yet small grains of unground black.

2. It must be of the proper strength (strong, middle, or weak); it must be suitable to the press (machine or hand) and must be readily transferred from the rollers to the type.

3. It should not clog up the type, and must wash off readily.

4. It should not dry too rapidly or too slowly, but in a definite time; in the former case it would dry upon the paper and type during printing, the paper would adhere to the type, would tear, and thus make further printing impossible; in applying the ink to the type, pieces would be torn out of the roller and would stick to the forme, an accident which cannot be allowed to happen. On the other hand, the ink must not dry too slowly, otherwise the printed sheets could not be taken off, folded, and sent away, which would be especially inconvenient in the case of newspapers.

5. When the ink is dry the colour should not rub off, otherwise the sheets in a pile would print off on one another and make reading impossible, also the hands of the reader would be soiled by the black ink, which has often been a trouble with newspapers.

6. The ink should have no strong smell, or the smell should disappear as the varnish dries.

7. The ink should not "run" after the print is dry, *i.e.* oily margins should not form round the letters. This should be especially the case with letterpress inks; it is not so important in the case of newspapers, which are printed, read, and may then be thrown away, although it should not occur even here.

Originally printers' varnishes were made exclusively from pure linseed oil or walnut oil, which was heated to a high temperature, almost to the flash point, when the oil became thick to an extent depending upon the duration of heating. For a number of years a different course has been followed, and now, in addition to thickened linseed oil, rosin, rosin oil, soaps, turpentine, tar oils, and even mineral oils are used for varnishes intended for the lower qualities of letterpress inks, especially for newspaper inks.

In the manufacture of oilcloth, leather cloth, and waterproof materials, thickened linseed oil is used, either alone or in combination with hard resins (copals). The fabric which forms the base of almost all such products is first covered with a priming layer, then follows the layer of paint, made by grinding pigments with linseed oil thickened by boiling; finally, 1 to 3 coats of a composition are applied, which essentially contains thickened linseed oil, together with copal, driers, and a diluent (turpentine or benzine). These relatively thin layers of paint and varnish are dried entirely at the high temperature of 50° to 90° C., according to the nature of the material and of the colour; on the one hand, drying in the air would require too long a time, and, on the other, the surface would not attain the requisite hardness, but would be liable to stickiness. The higher the temperature in the drying process, the more rapidly the various layers dry and the harder they become. Although in this manufacture considerable quantities of driers are used, yet they are not indispensable; for example, raw linseed oil treated with only 1 per cent. of a suitable metallic compound dries in a few hours at 50° to 60° C. Thickened linseed oil thus forms the basis for the preparation of all the materials mentioned. There are now several methods by which this substance can be made: heating over the fire, heating with superheated steam, and by blowing cold or hot air into linseed oil heated to a lower or higher temperature.

In heating over an open fire, the flash point lies tolerably near the temperature which is required for thickening; the linseed oil may thus very readily take fire, and it is necessary to work with great care. In using superheated steam, the danger of ignition is diminished, since the oil cannot run over into the fire, and the temperature is uniform, so that overheating cannot readily occur. The thickening of linseed oil by blowing cold or heated air into the gently heated oil is absolutely without danger, because a considerable rise in temperature does not occur; the process may be carried out between 135° and 150° C.

Somewhat divergent views are held in regard to the suitability of linseed oil, thickened in this manner, for the preparation of the materials before mentioned, which may be generally designated waterproof materials. On the one hand, it is maintained that the product is as good and as elastic as the substance prepared by the "boiling" process; on the other hand, it is stated that the oil when applied soon becomes brittle and the coating cracks. The author's view is that the latter evil only occurs when the oil has been unduly thickened, so that it requires too much diluent (benzene, benzine, turpentine) to be made capable of application—a condition which can be readily avoided if care be taken in the thickening process.

In the linoleum manufacture two different processes are used with linseed oil, which is thickened by heating or blowing in air, or is converted into a solid friable mass, which is then kneaded together with the remaining substances employed in the composition. In the latest process, solid substances: cork meal, wood meal, pigments, etc., are mixed with the linseed oil and the mixture then exposed to a current of hot air, by which the oil is then more quickly transformed into a solid product.

In regard to the thickening of the drying oils, Livache remarks: "In whatever manner the drying power of the drying oils is increased, there is obtained, under all circumstances, a product differing little in consistency from ordinary linseed oil. Frequently a thickened oil is required, either in order to shorten the time of drying, or in the preparation of varnishes, or finally, for priming coats, from which a thin oil might be too completely absorbed by porous material. The simplest way of thickening oil is to allow it to fall down from a certain height in thin layers, exposed to the action of the air; but this process is very lengthy, and the oxidation difficult, even in working at 50° to 60° C., because when the oil has thickened to a certain extent it is no longer fluid enough to flow in a thin stream. In addition, a portion of the oil adheres to the surface upon which it falls, thickens or dries, and thus causes a considerable loss when a quite solid product is not desired. Finally, a skin forms on the oil when the thickening has proceeded to a considerable extent. The skin prevents the oxidation and thickening of the oil beneath it. Endeavours have been made to increase the fluidity of the oil by adding a suitable solvent. If, for example, equal parts of linseed oil, boiled with manganese borate, and benzine are treated in a closed vessel with air, a rapid absorption of oxygen takes place, which is still more rapid at 40° to 50° C. If the air be renewed by adding sufficient oxygen to produce complete oxidation of the oil (at least 15 to 16 per cent. of the weight of the oil) the mixture thickens, and when finally the benzine is separated by distillation a sticky mass remains. If the treatment with oxygen be carried still further, there is formed linoxyn, which is insoluble in benzine, and is left as a dry and very elastic substance when the solvent is distilled off.

"In practice at the present time the oil is frequently boiled 3 to 4 days at a temperature of 250° to 300° C. A thick or "strong" oil is then obtained, which may be made less viscid by the addition of a little linseed oil boiled 6 to 8 hours at 200° C. This boiling at such high temperatures has the disadvantage that free fatty acids are formed, which require a long time to oxidise and become solid. In England and Germany it is preferred to boil the oil with steam or heated air in pans of large diameter and small depth, so that the air above the oil may be continually changed; deep closed pans are, however, also used. In certain works, especially in Holland, no driers are added to the oil, but a current of hot or cold air is led through it; the product is of good quality, but the operation is somewhat too lengthy.

"For certain purposes the boiled and thickened oils are stored in copper or iron tanks, which should be enamelled or covered with a very thin plating of silver on the inside in order to avoid discoloration of the oil."

The thickening of linseed oil for printers' varnishes, stand oil, varnishes used for oilcloth and patent leather, is a process of distillation conducted in the presence of air, to which Mulder had already given attention.

All fatty oils are decomposed by heating above 250° C.; below this temperature they are little changed, above it the change is greater the higher the temperature. The products of the decomposition of neutral fatty oils, drying or non-drying, contain acroleïn, a decomposition product of glycerin, thus free fatty acids must also be formed. If the neutral oil contain palmitin, palmitic acid may be expected in the distillate. When free palmitic acid is heated, it distils partially unaltered, but is partially decomposed at higher temperatures into palmitone, so that carbonic acid and water are set free. Since palmitin occurs in linseed oil, among the products of the dry distillation of this oil at a high temperature, carbonic acid, water, palmitone, palmitic acid, and acroleïn are to be expected. These substances are evolved when linseed oil is boiled, and in larger quantity the longer the boiling is continued.

By boiling linseed oil exposed to the air, boiled oil is first obtained; if the oil be further boiled at a high temperature a soft mass forms on cooling, by further boiling it becomes still thicker, and when brought upon paper produces no grease spot. The vapours evolved from the oil in this decomposition generally take fire, but this phenomenon is not indispensable. When boiled beyond a certain point linseed oil no longer dries, but becomes sticky or elastic. If it is to retain its drying properties it must be heated until the linoleïn begins to decompose (Mulder); when this occurs boiled oil begins to pass over into varnish. Among the first substances which are driven off on boiling are the decomposition products of oleïn, myristin, and palmitin.

If linseed oil be heated until it becomes thick, and then boiled with dilute nitric acid, it turns solid. The separation of the anhydride of linolic acid from the glycerin is caused by the nitric acid, so that the smell of acroleïn becomes perceptible. Finally, the mass becomes like caoutchouc (according to Jonas), it no longer sticks to the fingers, cannot be melted, and is dissolved to an emulsion by carbon bisulphide. If this caoutchouc-like mass be boiled in strong caustic potash the two substances unite, but the former is not dissolved, and acids again precipitate it. It is also dissolved by alcoholic potash and reprecipitated by acids. In ether containing alcohol it swells, dissolves in more ether, and is again precipitated by alcohol. It swells up in mineral oil, but is not dissolved; a large quantity of turpentine dissolves it.

Mulder examined the products of the dry distillation of linseed oil; an account of his work follows:—0·5 litre of linseed oil was heated almost to boiling in a retort, provided with a receiver, in a sand bath by a gas flame. The retort and its neck were filled with air, but the air did not become hot. In the first 6 hours there distilled a mixture of substances slightly coloured and quite fluid, with a smell of acroleïn, afterwards a white buttery mass came over; the quantity of the two together was $\frac{1}{20}$ of the oil.

In the second 6 hours the distillate was caught separately; it was a white buttery substance with a slight smell of acrolein, equal in quantity to about 5 per cent. of the original oil. The contents of the retort were now quite thick.

In the third 6 hours the same buttery mass distilled over at the same temperature, but in decreasing quantity, which was finally so small that the distillation was interrupted after 36 hours. Distillation at a low temperature, below the boiling point of the oil, by heating slowly for 36 hours to complete the distillation of 500 grms. of oil, is necessary to obtain a good result. The hydrocarbons are thereby reduced to a minimum, the fatty-acids of two of the glycerides are obtained in appreciable quantity, and no trace of carbon is perceptible. The volatile portion is almost colourless.

We will now consider the residue in the retort, the characteristic product of linseed oil, and apparently of all drying oils, since it is the most important for our purpose. This non-volatile portion is almost free from fats, yet, as a precaution, it was extracted with ether to remove any residual fat, and in fact some portion dissolved. The residue, insoluble in ether, was dark in colour when seen in the mass, but in thin layers appeared straw yellow; it was very elastic, and quite similar to caoutchouc. Elementary analysis gave the following results:—

	Found.		Calculated for
	(I)	(II)	$C_{32}H_{54}O_3$
C	79·1	79·2	79·0
H	11·2	11·2	11·1
O	9·7	9·6	9·9

It is thus remarkable that the caoutchouc-like mass which forms the residue of distilled oils is the anhydride of linoleïc acid, $C_{32}H_{54}O_3$. This caoutchouc-like substance is converted by nitric acid at the ordinary temperature to a reddish-brown resin. Ammonia does not attack it, it remains quite insoluble; in caustic potash solution it becomes suspended on heating, and is quite dissolved on the addition of more water to a clear colourless solution, from which hydrochloric acid precipitates a substance, which after many hours appears as a sticky layer upon the surface, soon changing to an elastic skin.

For further examination the elastic skin was removed from the surface of the liquid, washed with water, dried in the air, and treated with ether. The ethereal solution gives on filtering only traces of insoluble matter. When the solution is evaporated a resinous substance remains, deep in colour; it was again dissolved in ether, and the solution decolorised by filtering through animal charcoal. After evaporation of the ether, the residue was dissolved in boiling absolute alcohol, in which it was slowly taken up; on cooling it separated to a great extent as a coloured resin. In external properties this substance differed little from the original, it remained sticky even after many months' exposure to air, and might thus be assumed not to be further oxidised. From the commencement of the distillation a considerable quantity of acroleïn was evolved; it was collected with the volatile products, which at first formed a colourless liquid partly soluble and partly insoluble in caustic potash solution. The hydrocarbons and fatty acids which were formed were not further investigated. The substances which were not volatile below 100° C. consisted of several acids. The portion which was volatile at the ordinary temperature was completely dissolved in a solution of sodium carbonate on heating, carbon dioxide being evolved, a result which was not obtained if the linseed oil was not distilled at a very low temperature.

The whole of the substances not volatile below 100° C. were extracted with warm water until nothing more dissolved. From this water a crystalline deposit separated, which was again dissolved and recrystallised; it was sebacic acid. The acroleïn which remained was already changed into acrylic acid; the liquid had almost lost the smell of acroleïn.

If we examine the results of the dry distillation of linseed oil at a low temperature (near the boiling point), we find, as residue in the apparatus, linoleïc anhydride, and as the volatile distillate, acroleïn (partially oxidised to acrylic acid), sebacic, palmitic, and myristic acids.

THE MANUFACTURE OF THICKENED LINSEED OIL (BURNT OIL, STAND OIL) BY HEATING BY FIRE OR BY SUPERHEATED STEAM, AND BY A CURRENT OF AIR

As we have already seen at the commencement of this section, linseed oil may be thickened in various ways:—

1. By heating over an open fire.
2. By heating by means of superheated steam.
3. By blowing cold or heated air into the oil at higher or lower temperatures. The difference between the methods employed for printers' varnish and for oil to be used in the manufacture of oilcloth, waterproof materials, and linoleum, is simply, that for the first purpose the oil is boiled without driers, for all the others with driers. By the first process there results by a relatively long operation a pale product, in the second process in a shorter time dark well drying products are obtained. By blowing in air there is produced, according to the temperature, pale or dark varnish, but under all circumstances and by all three processes dark varnish results when the heating or blowing takes place in

the presence of driers. Varnish of the same drying power, but paler in colour, is obtained when the thickened oil is allowed to cool to 130° C. and the drier then introduced.

The apparatus used in thickening linseed oil by boiling is an iron pan, of greater depth than width, erected in such a manner that the flames reach only half-way up the pan. The upper half is heated neither by fire nor the fire gases. The pan may be quite open, provided with a lid through which the vapours are carried away by means of a flue to the open air, or it may be quite closed, after the manner of a retort. Several of these pans, as also the installation for blowing air through oil, are described at the conclusion of this section.

In order to produce thickened linseed oil by any of these processes successfully, that is, within a reasonable time, it is absolutely necessary to use good tanked oil and to subject it to a preliminary operation before the real thickening. Any linseed oil may be thickened by great heat, but there is too large an expenditure of time and fuel, and the principle of every process should be to reduce the cost to the smallest possible limits.

Good tanked linseed oil is brought into the pan, so that it is filled to within 10 to 15 cms. of the edge; it is then slowly heated. At first the temperature is maintained at 130° to 140° C., when the oil froths, though this is not the case if it is free from moisture. After about an hour, when the frothing is over if it occurs, a very powerful fire is made and the temperature raised to 270° C. The colour then changes from yellow to pale green, and slimy lumps may form in the oil, but should not in a good oil sufficiently tanked. When this point is reached, the fire is allowed to die out and the oil left at rest for 24 hours, so that all the separated matter may sink to the bottom. The clear oil is then run or drawn off and brought into a second pan, or, in case a second is not provided, into other vessels, the foot removed (to be used for paints, cements, etc.), the pan carefully cleaned, and the oil again introduced. A very powerful fire is now applied to this prepared oil, so that in a short time a temperature of 270° to 300° C. is reached. Thick and pale linseed oil is only to be obtained by rapid heating to the requisite temperature; slow heating produces a darker product, at the cost of much time and fuel. When the temperature reaches 300° C. a more gentle fire is used until it reaches 350° to 360° C., at which the oil is maintained during 1 to 2 hours. The evil-smelling vapours which are evolved, which cause coughing, are allowed to escape into the air or are drawn off by the hood, let down over the pan, into the chimney or the open air. After the lapse of 2 hours, during which the oil is at 360° C., the temperature is slowly and carefully raised to 375° or 380° C., above which it is not advisable to go, since the oil may catch fire and continue to burn at a higher temperature. This is the critical point in the preparation of thickened oil, and demands care and attention. The time during which the oil must be maintained at this temperature varies from 2 to 4 hours, according to the nature of the oil; from time to time test portions are withdrawn and rapidly cooled, in order to see whether the oil is sufficiently thick. When a portion, taken between two fingers, may be drawn out without breaking into sufficiently long threads, the process is finished, then the fire is drawn, the doors opened, and the high temperature of the oil reduced as quickly as possible in order to avoid danger of fire. The time required to produce oil of varying consistencies cannot be given, the test portions can alone decide; we can only state the general rule, that the longer the oil is maintained at the high temperature the thicker it becomes. If, in spite of all care, the oil should take fire, the cover hung from the roof by chains (not ropes) is let down, and by opening the fire doors and drawing the fire the oil is cooled; the temperature must sink considerably before the cover is again removed, since the sudden entry of air to the hot oil might cause it again to take fire. When the oil has attained the proper consistency it is allowed to cool for some time and then brought into the storage vessels.

In working with driers, and especially with those containing lead, the oil rapidly darkens even at 240° to 250° C., and on further heating becomes covered with a reddish-brown skin, which must be carefully pushed back to the sides of the pan or the oil will easily take fire. However, it is far more advisable, in order to obtain a pale varnish of the same drying power, to thicken the oil without driers to the desired consistency, and then, when the temperature has receded to 130° to 140° C., to stir in the driers. The varnish will be far more durable, and remain elastic longer, if the driers are not boiled with the oil.

In boiling oil in closed apparatus, whether by fire or superheated steam, there is considerably less danger of fire. Thermometers must be placed in the apparatus, so that the temperature may be accurately known, and overheating cannot occur.

The consistency of thickened linseed oil varies with the purpose for which it is to be used. Printers require varnish both thick and thin, as also the manufacturers of oilcloth and waterproof materials. In order to obtain a certain consistency it will often be necessary to mix thick and thin varnish. A consistence measurer is the best instrument for an accurate determination of the consistency; such an instrument, which is hardly required by the experienced varnish boiler, is shown in Fig. 33. It measures the time required for an air-bubble to rise through the oil in a glass tube. A cylindrical glass vessel, which may be closed by a carefully ground stopper, is filled so far with the oil to be tested that

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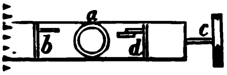

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means of superheated steam. The pan should not be too deep or the air current will lose a portion of its action; on account of the resistance which the oil offers to the free passage of the air, when the pan is too deep the air may not be able to overcome the pressure due to the height of the column of oil. Thus, even when a powerful current of air is used, the depth of the oil in the pan should not exceed 80 cms.

The thickening of drying oils by means of a current of cold air is a tolerably simple operation, free from danger even when high temperatures are used; however, 270° C. is never exceeded, and generally much lower temperatures are used. The process with heated air is somewhat more complicated, and has no particular advantage; the introduction of finely powdered driers with the current of hot or cold air may be regarded as purposeless, for the reasons previously given. The driers may be introduced with the same effect when the thickened oil has cooled to 130° to 140° C.

The simplest arrangement for blowing oil, which may be introduced into any pan, is a ring-shaped iron pipe which rests by small feet upon the bottom of the pan, and communicates by a vertical tube with the blowing machine. The ring-shaped pipe should be 5 cms. in diameter, the holes situated in the lower part of the pipe should be 0·5 mm. in diameter. The entrance of air into this pipe may be regulated by means of a tap. The exit holes for the air should be pointed downwards, because, in spite of the great pressure of the air produced by the blowing machine, an upward current does not produce the desired effect in practice. The oil is kept in violent motion by means of the downward current of air, so that it rises in the form of spray above the edge of the pan; from holes in the upper surface of the ring the air might often not be able to reach the surface of the oil. Bends in the pipe carrying the air should be avoided as far as possible, so that the current of air may be little retarded; when practicable, it should travel in a straight line. The blower or fan must have sufficient power to yield a sufficient quantity of air; regard must be had to the distance and the height of the column of oil to be overcome. The pan itself should be provided with a removable cover, which can be let down when the air is forced through the oil, in order to avoid spraying or to return the oil to the pan.

In regard to the operation of blowing itself, it is carried out as follows:—The oil is brought in the pan to a temperature of about 110° to 120° C., the tap for the air supply is then opened slowly and cautiously, so that the full pressure may not come at once into the pan; the quantity of air is then regulated by means of the tap according to the effect observed in the pan. The oil should not be thrown out of the pan, but should form a spray above the surface. The temperature is now somewhat reduced; even if the fire should be quite extinguished, only a difference of 15° to 30° will be produced, since the friction produced by the passage of the air through the oil develops heat. Thus, in order to blow at 110° to 120° C., the fire will require little stoking, and it is easy to maintain the temperature at this height. The time required to obtain an oil of syrupy nature varies greatly, and may reach 2 to 4 days. At first, and even after 1½ to 3 days, no change, or very little, can be observed in the consistency; towards the end of the operation, after the introduction of considerable quantities of air, the alteration becomes evident, and the thickening takes place rapidly. All oils which are blown at a low temperature remain pale; they become even paler than the original oil, and, for example, may be used for the finest white oilcloths. As the temperature of the oil rises, the time required for thickening becomes less, but the oil darkens considerably. If the oil is blown together

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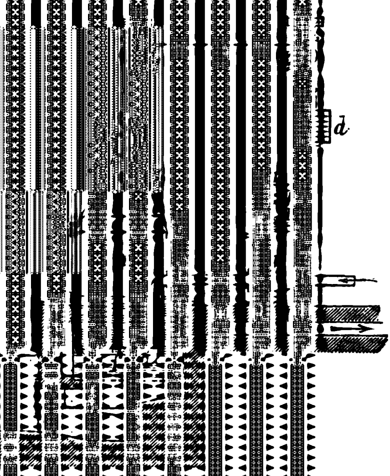

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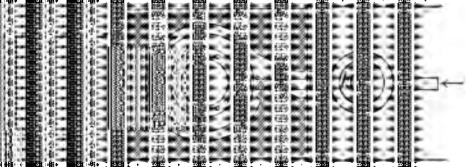

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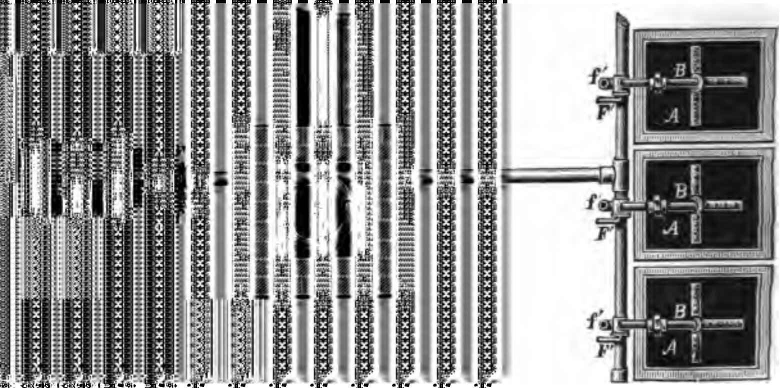

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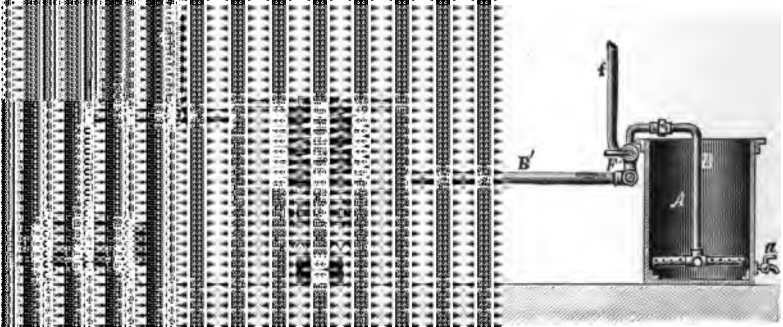

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of the pipe *f* is perforated, so that the air forced in by the blower emerges in numerous fine jets. Between the blower and boiler a small pan *h* is introduced, suspended in an air-bath *i*, and heated by the waste heat of the boiling pan or by gas. The cover of this pan carries a vertical pipe with two taps k_1 and k_2, between which is a spherical vessel *l*. Above k_1 is the dish *m*. This apparatus is used to introduce powdered driers (oxidising agents) into the air current. This is accomplished by filling the dish *m* with the material and allowing it to fall down through the open tap k_1 into *l*. On closing k_1 and opening k_2 the powder falls down into the air current, by which it is carried into the oil. The boiling pan is partially filled with the oil to be oxidised, and whilst it boils air, charged with the oxidising powder, is forced into it in fine streams. The oil is then changed in 15 to 18 hours. Towards the end of the blowing the oil suddenly thickens.

Figs. 36 and 37 show the plan and vertical section respectively of an apparatus for treating linseed oil with heated air. A, A, A is a series of tanks which contain the oil to be treated, each is provided with a pipe B, by means of which the hot air is introduced. This pipe ends in branches, suspended immediately above the bottom of the tank, without touching it. The branch pipes are perforated, so that the air is forced through the oil in fine streams. The pipes B, B, B of the various tanks are connected with the pipe B′, which is supplied with heated oil from the coil C of the heater D.

The coil C is connected with a compressor E (or Root's blower), and thus obtains a continuous supply of air, which is regulated by means of the valve T. G is a loaded safety valve. The branch pipes B, B, B are also provided with taps, which regulate the supply of air to the individual tanks, and by which the air can be cut off when the process in any one tank is finished. The cocks F and F′ are 3-way; their construction is shown in Fig. 38. They are so made that the whole or a portion of the air may be blown off into the open. If all the tanks are in use the maximum supply of air is required. When the air becomes heated to about 312° C. the cock F must be partially opened. But if the temperature of the air in the coil rises above 312° C. the tap must be opened wider, in order to send a smaller quantity of air through the outlet *f* and a larger quantity into the coil. Thus more air goes into the heater, and the oil is prevented from burning. The temperature of the oil should never rise above 205° C. The process should be conducted so that the oil in the different tanks is in different stages of treatment, so that when the operation is finished in one tank the treatment of the oil in another may be commenced, until the whole has been treated. When the oil in any one tank is finished, its tap F′ is turned in order to cut off the hot air and to prevent the temperature of the oil in the other tanks from rising unduly. For example, if the tanks contain 227 litres of oil, so much hot air is introduced that the temperature rises to about 121° C., which is maintained for about 5 hours and then raised to 205° C. Care must be taken that this temperature is not exceeded; it is maintained for about 5 to 6 hours, in order to drive off the acrid vapours. The regular

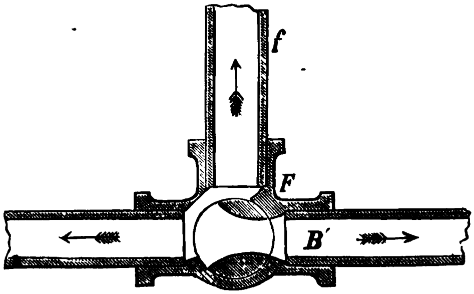

Fig. 38.—Apparatus for thickening Linseed Oil by means of Hot Air. 3-way Cock for distributing the Air Current.

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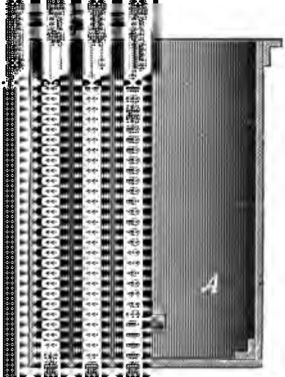

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arranged so that the liquid forced up by the pump may leave it in jets. The oil is heated in the pan to the desired temperature, then drawn off by the pump and forced through the pipe and the holes of the box. It leaves the box in fine streams, comes in contact with the current of air in the cylinder, and falls into the first pan, from which it runs through the connecting pipe to the second pan. The oil is moved through this circuit until it is sufficiently oxidised and thickened.

FORMULÆ FOR PRINTERS' VARNISHES WITH ROSIN AND LINSEED OIL

	Parts by weight.		
	Weak.	Medium.	Strong.
Rosin	12·5	12·5	12·5
Stand oil	50	50	50
Rosin soap	1·5	1·5	1·5
Stand oil (very weak)	3·5	2	...
Rosin	25	25	25
Stand oil	50	50	50
Rosin soap	5	5	5
Stand oil (very weak)	4·5	3	...
Rosin	38·5	38·5	38·5
Stand oil	50	50	50
Rosin soap	3·5	3·5	3·5
Stand oil (very weak)	6	4·5	...

The principal constituent of these printers' varnishes is the thick boiled linseed oil (stand oil). It is not boiled long enough to be of the thickness required for the various strengths, but still so long that it forms no grease mark on paper. In this case rosin alone is used as the thickening agent, since it has been found to produce a varnish of the same strength as a varnish from pure linseed oil, but much cheaper, so that it can be used for cheap printing inks. The oil is first boiled, allowed a short time for clearing, and then, after reheating, the rosin is added, as free from water as possible. The rosin is broken into small pieces in the vessel in which it is to be melted, and then fused over a slow fire. When it has become quite limpid, and has given off some vapour, the rosin soap cut into small pieces is added, and when that has dissolved, the boiled linseed oil (stand oil). The whole is well stirred, left half an hour on the fire until the whole is quite fluid, and then filtered through fine linen to remove all the impurities of the rosin. The finished varnish is allowed to stand as hot as possible, so that all small particles of impurities still present, which would not sink in the thick varnish, may deposit. The bright varnish is drawn off after a few days.

X

FORMULÆ FOR LINSEED AND ROSIN OIL VARNISHES

Weak varnish with stand oil.

	Parts by weight.		
	a.	*b.*	*c.*
Stand oil (very weak) .	7	9	12
Rosin soap	3	5	7
Rosin oil	50	50	50
Rosin	25	50	50
Stand oil	50	50	50

Medium varnish with stand oil.

	Parts by weight.		
	a.	*b.*	*c.*
Stand oil (very weak) .	4	6	9
Rosin soap	3	5	7
Stand oil	50	50	50
Rosin oil	50	50	50
Rosin	25	50	50

Strong varnish with stand oil.

	Parts by weight.		
	a.	*b.*	*c.*
Rosin soap	3	5	7
Stand oil	50	50	50
Rosin oil	50	50	50
Rosin	25	50	75

Weak varnish with raw linseed oil.

	Parts by weight.	
	a.	*b.*
Thick turpentine (crude)	5	...
Rosin soap	5	7
Linseed oil	13	35
Rosin oil	24	95
Raw linseed oil	21	100

Medium varnish with raw linseed oil.

	Parts by weight.	
	a.	*b.*
Rosin soap	5	7
Thick turpentine (crude)	5	...
Linseed oil	105	90
Rosin oil	210	240
Rosin	210	215

Strong varnish with raw linseed oil.

	Parts by weight.	
	a.	*b.*
Rosin soap	5	7
Thick turpentine (crude)	5	2
Linseed oil	87	25
Rosin oil	240	80
Rosin	210	100

In the preparation of these varnishes the rosin and rosin oil are heated together in a pan over the fire, then the soap is added, and finally the linseed oil. The mixture is then maintained at 120° to 140° C. for some hours, so that the smell of rosin oil may as far as possible be driven off and the materials intimately mixed.

VARIOUS PRINTERS' VARNISHES

For best illustration inks.

		Parts by weight.
(1)	Copaiba balsam	70
	Linseed oil	50
	Rosin	110
	Benzoïn	3
	Balsam of Tolu	2
(2)	Copaiba balsam	85
	Linseed oil	40
	Rosin	115
	Benzoïn	3
	Balsam of Tolu	2

According to Gogneau.

		Parts by weight.
(1)	Linseed oil	979
	Rosin	735
	Syrup	245
	Litharge	12·5
(2)	Linseed oil	400
	Rosin	980
	Syrup	490
	Litharge	6
(3)	Linseed oil	980
	Rosin	958
	Syrup	980
	Litharge	12·2

The oil is mixed with the litharge and a slow fire kept under the pan until the oil begins to boil and the froth to disappear. The rosin is now melted with a small quantity of linseed oil and added to the oil in the pan, when the froth disappears. The mixture is well stirred, allowed to cool partially, and the syrup finally added.

According to Roöl.

	Parts by weight.
Thick turpentine (crude)	9
Soft soap	10
Oleïn	4

According to Pratt.

	Parts by weight.
Rosin	390
Rosin oil	500
Yellow soap	90

The thickening of drying oils, Blakemann's process. —A mineral soap, and by preference one which has the power of absorbing carbon dioxide, such as an alumina soap, is dissolved, by the aid of heat, in a vegetable or animal oil which is not decomposed at the temperature employed. Of the vegetable oils, sunflowerseed oil is suitable, but cottonseed oil is preferable. About 20 to 30 per cent. of the mineral soap is added to the oil, or so much that the mixture is gelatinous. The soap may also be added to complete saturation, but the above quantity is preferable. The oil thickened in this manner is now mixed with the drying oil to be treated, either simply by stirring or by gentle heating on the water-bath. The added quantity of the oil thickened by the mineral soap may amount to 5 to 7 per cent. of the drying oil, or more or less. When the mixture has cooled it is a far better vehicle for pigments than before treatment. The colour of the oil is not injured by the process, whilst its drying properties remain unimpaired or are increased. The reason being that, on the one hand, the high temperature required completely to dissolve the metallic soap does not injure the cottonseed oil, but rather tends to exert a bleaching action upon it, and that, on the other hand, the characteristic affinity of the mineral soap for carbon dioxide favours and increases the drying action. In this process the mineral soap may not be mixed directly with a drying oil for the purpose of thickening it or to obtain a vehicle for pigments. Such mixtures have been previously made, but only with partial success, since the high temperature required to produce a perfect mixture causes the oil no longer to be capable of being bleached. This difficulty is avoided by the present process, since the mineral soap is first mixed with an oil which is not decomposed at the temperature required, and this mixture, in which the oil has been made basic or alkaline, is added to the drying oil. The colour of the drying oil is thus preserved, and if the mineral soap is capable of absorbing carbon dioxide the drying power of the oil is at the same time increased. The drying oils treated in this manner, such as linseed oil or similar oils, may be kept for later use or at once mixed with pigments for use in paints. If necessary, any of the ordinary commercial driers may be added to the paint.

Linseed oil for the manufacture of linoleum must be treated in quite a different manner to that required in the preparation of printing inks, oilcloth, etc. The substance used for the latter purposes is a thick mass, still homogeneous, which can be liquefied by heating or by the addition of turpentine, benzine, or other solvents, and which dries to a more or less colourless adherent skin when applied in thin layers. On the other hand, in the preparation of the drying oils for the manufacture of linoleum, the condition is sought, which the oil has after drying in thin layers, *i.e.* a soft elastic dry skin, capable of being kneaded with solvents, resins, cork or wood meal, to form a mass which can be rolled between rollers into sheets with or without a backing of fabric.

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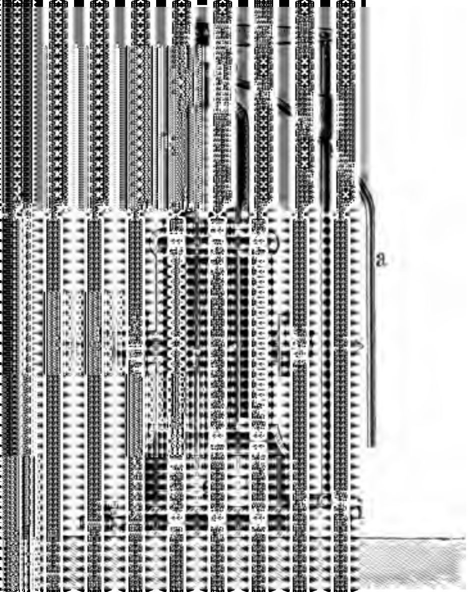

Oxidising

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in by a fan. Two sides of the column are formed of glass, so that light may enter in order to bleach the oil; *f* and *f′* are perforated zinc plates, *f* is intended to distribute the air, as it enters the apparatus, through the whole of the column; *f′* forms the air exit and retains the particles of oil. A rapid current of air is not necessary, it is only required to renew continuously the air in the apparatus. The oil falls into the tank *g*, below which is the space *h* into which steam is admitted at a temperature of 100° to 177° C. The higher the temperature the more rapid is the transformation of the oil. By means of the pipe *i* the oil is again brought to the pump, which is not shown in the illustration. The pump again forces the oil into the tank *b*; the process is continued until the oil is sufficiently changed. At the upper end of *b* is a small cylinder, in which is a piston connected with the lever *l*, loaded in accordance with the pressure to be maintained in *b*. The rod *m* is connected with the tap on the pipe *i* in such a manner that, when the pressure in *b* becomes too great, the piston rises, thus closing the communication between the pump and the tank *g*.

In the second apparatus constructed by Walton, linseed oil mixed with the oxidising substances is heated under constant stirring in an open pan over a fire; a chain pump *b* (Fig. 41) raises the hot oil into a spout *c*, from which it runs into the trough *d*, running along the wall of a large chamber K (the oxidising chamber), roofed with glass. In front of the outlets of the trough are wheels *e*, 500 mm. in diameter, upon which the boiled oil falls in a thin but wide stream. The wheels move at the rate of 600 revolutions per minute, and break up the oil into fine drops, which are thrown into the chamber, where they slowly sink to the bottom. This rain of oil flows in the opposite direction to an air stream driven into the chamber through the pipe *g* by the fan *f*, and escaping through openings *h* in the wall, which are at the

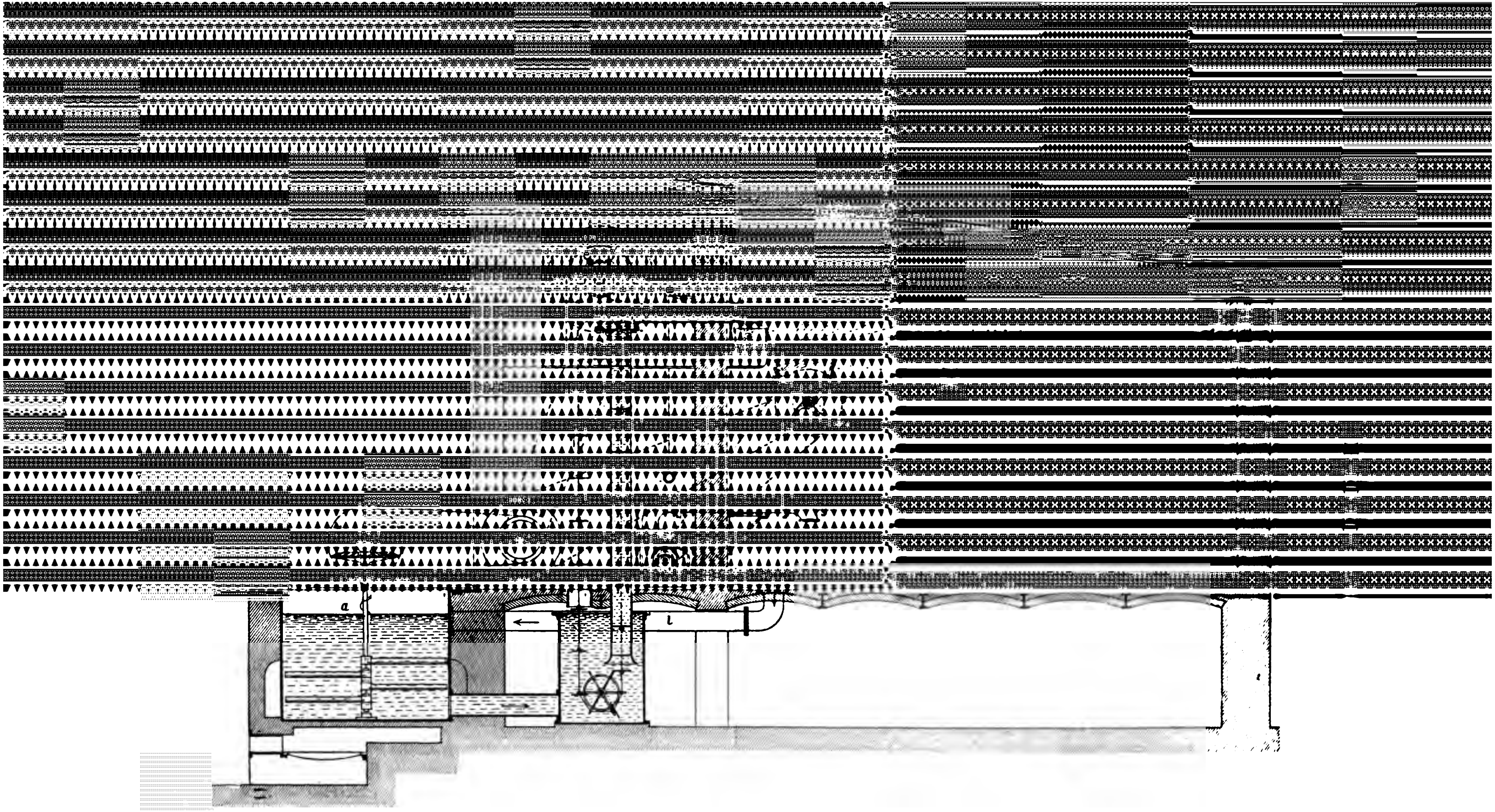

FIG. 41.—Walton's Apparatus for Oxidising Linseed Oil.

greatest distance from the entry. These openings can be wholly or partially closed by valves *i*, which thus regulate the strength and direction of the air current. A layer of gauze *k* prevents the oil spray from passing through with the air. The oil collects at the bottom of the chamber and flows back to the boiler *a*, through the pipe *l*; the series of operations is repeated until sufficient oxidation has taken place.

The linseed oil which has been prepared simply by boiling, or by boiling and treating with air in the apparatus just described, is now spread out in very thin layers upon a stretched fabric and exposed to the action of a current of hot air. Walton has also devised various arrangements for the performance of this operation. The oil is first brought into cube-shaped vessels with an edge 90 to 100 cms. long; into these vessels fit wooden frames in which iron rods are placed above and below at certain distances and always opposite one another. A cotton fabric is now wound over these iron rods, the end of the piece being fastened in a suitable manner, so that a certain number of lengths, corresponding to the number of iron rods, are placed near together. The length of the fabric is adjusted so that the end is again fastened to the last rod. The frames carrying the cotton are now immersed in the linseed oil, in which they remain until the fabric is saturated, when they are taken out, hung up over the vessel to drip, and then exposed to a current of hot air, which converts the linseed oil in about 24 hours to a dry mass, no longer sticky. After drying, the frames with the fabric are again immersed in the oil and again dried, this alternate dipping and drying being continued until the layer is several millimetres thick. The vessels of linseed oil are placed in a row, the frames are dipped into them simultaneously by a mechanical arrangement, taken out at the same time and fastened at a certain height, so that manual labour is entirely avoided.

In another arrangement of the oxidising apparatus the fabric is fastened to iron rods, so that it hangs down lengthways. Above the fabric a trough rests upon rails; it ends in boxes in which are narrow slits parallel to the supports of the fabric. The oil is pumped into the troughs, runs from these into the boxes, and thence through the slit on to the fabric, the whole length of which it traverses, and finally collects in a well. The oil is again pumped into the tank, and when the fabric is dry the oil is again led over it. This process is repeated until a layer of dried linseed oil about 1 cm. thick is obtained, which naturally requires several months.

The fabric is then rolled upon drums, and the layer of linseed oil removed by a peculiar arrangement consisting of rollers heated by steam, through which the end of the fabric is passed after the layer of dried oil has been removed for a length of about 10 cms. In consequence of the position of the rollers and the pressure they apply, the solid coating is stripped off, the fabric passes alone through the rollers and may be again saturated with oil. In the case of very thin material, which does not possess the necessary strength to be used a second time, the dried oil is not removed, but fabric and oil together are ground up and added to the linoleum mixture. The end product of Walton's process, when removed from the fabric, is a plate 8 to 10 mm. thick, which, in accordance with the periodic application of the oil, shows distinct stratification in section. In layers of 3 to 4 mm. thickness the mass is no longer transparent, and appears of a dark orange-red colour. In thinner layers the colour is paler; a layer 1 mm. thick is yellow with a tinge of brown. The mass is completely homogeneous, not sticky, and similar to indiarubber, like which it is very elastic, but is far less soft and exposes less resistance to tearing. The mass is so wanting in coherence that it can be ground in a mortar to a yellowish-white powder, which coheres with difficulty and then not permanently. It has a slight sweetish odour, similar to that of fresh linseed oil, but much milder; it is free from taste. When heated it remains solid, and may be ground to powder; under powerful pressure it coheres permanently.

The process of Hertkorn, for the rapid oxidation of the drying oils, depends on the observation that the oxidation takes place much more rapidly when the oils, with or without driers, are spread upon, or mixed with, voluminous powdered materials, light in weight, through which hot air is led. The most suitable substance for this purpose is the mixture of cork and wood meal used for linoleum. In this process the oxidation is complete in 1 or 2 hours, whilst for the methods previously used days and even months are required. The linoleum mass obtained by the new process is quite free from smell, thoroughly uniform and completely oxidised, also elastic to a high degree. The mixture of the oil with the fillers is the most intimate obtainable. In order to increase the binding power and the elasticity, certain hard resins are added in fine powder to the semi-oxidised mass. Varying quantities of the oxides of the alkaline earth metals, or salts of these metals with weak acids (soaps), are added to the linoleum mixture in order to prevent the spontaneous combustion of the mass saturated with oil. These additions impart additional hardness to the linoleum, with a velvet-like smoothness, leathery pliability, and a toughness hitherto unobtained.

CHAPTER VI

BEHAVIOUR OF THE DRYING OILS AND BOILED OILS TOWARDS ATMOSPHERIC INFLUENCES, WATER, ACIDS, AND ALKALIS

THE drying oils, of which linseed oil is the only representative used on the large scale, and the boiled oils obtained from them, are rarely used alone in painting and decorating; linseed oil is, however, so used as a priming for iron and on woodwork, so that the natural colour of the wood remains. A far more extensive use is in intimate admixture with pigments for painting the most varied materials—wood, metal (principally iron), walls (plaster), stone, and linen. Paint is used with the object—(1) of providing the material with another colour, either for æsthetic reasons or to hide the subjacent materials; and (2) in order to protect the material from external influences—rain, moisture, and air. If the woodwork in buildings, such as of windows, doors, etc., is not protected by paint it will be injured by the action both of wet and summer heat, will quickly lose its pale colour, and separate into fibres. In the presence of much moisture, especially in such a position that it cannot dry quickly when wetted, it will soon be destroyed by rot and mould. If iron is not painted it will be rapidly rusted by the action of the atmosphere. The formation of oxide, when rust has once appeared, spreads so rapidly by means of carbonic acid and water, which are retained by the porous rust, that thin sheet iron is rapidly rusted through, and may even quite fall in pieces. Unprotected plaster eagerly absorbs rain and mist; certainly, it will soon dry again, but by the continued change from dry to wet it is injured, shells off, and finally falls away. All these disadvantages are to a certain extent obviated if the surface be covered with a layer of a drying oil or of a paint made from a drying oil, and this layer be then exposed to the action of the weather after an interval which depends on the position of the object, especially in regard to the incidence of sunlight. The principal reason for painting all objects is to protect them from the action of atmospheric influences; we must at once consider the effect of these influences on coatings of boiled linseed oil or of paint made from it. From the changes undergone by the very thin dry layers of the drying oils and paints containing them, we know that the protection afforded by these coatings must be relatively brief. Linseed oil, boiled oil, and oil paints change, when withdrawn from all other influences, by a further absorption of oxygen from the air and by the action of the sun's heat. Free linolic acid is further oxidised, as also unaltered linolin, and finally the end-product also begins to decompose and the coating then perishes. This is the fate of any coating of a drying oil or boiled oil, with or without pigments; it occurs in a longer or shorter time according to the nature of the material. Wood, plaster, and stone are of an open nature, they have pores and interstices between the particles, in which part of the paint is absorbed whether it contains oil alone or oil and pigment. Thus, on the one hand, an intimate combination is established, and, on the other, the drying oil in the pores remains undecomposed longer than that on the surface. With iron it is different, the metals have no pores, nothing can be absorbed. The paint remains on the surface, and thus it comes about that paint on iron-work has generally a briefer duration than on wood, plaster, or stone.

In addition to the action of the sun, other influences must be considered, these are—(1) rain, fog, and snow; (2) the movement of the air; (3) dust and sand, which are thrown against the paint; (4) the gases contained in smoke; (5) the pigment with which the boiled oil is mixed to produce the paint.

As we have previously seen, changes occur in the dried layers of oil; the coating, which was at first elastic, gradually becomes less elastic and more friable, finally forming a hard mass which can readily be scraped off. With the further lapse of time the layer decomposes still further, the union between pigment and oil is dissolved, and the pigment is removed from the groundwork by rain, wind, sand, and dust, which are driven against the object. There is no permanent protection; the practical duration of a coating of oil-paint upon wood and plaster, exposed to the weather, is taken as 5 to 10 years at a maximum, and on iron and other metals, also exposed to the weather, as 2 to 5 years at a maximum.

The influence of rain upon paint is inconsiderable, if the water does not stand on it for a long time; according to the author's experience, the diffusion of water through a coat of paint, assumed by Simon, does not take place. Simon supported a skin of paint, removed from its original position, in such a manner that it formed a hollow, in which water was placed, which after some time appeared on the under surface of the paint-skin in drops. This experiment, however, proves nothing, for there is a very considerable difference between paint attached to the surface beneath and a layer removed from this surface. The water, deposited by rain and snow upon a coating of paint, generally evaporates so rapidly that it would not have time to permeate the paint even if the latter were permeable. Thus there can be no question of the destructive action of water when it is not continued for a long period. The gases given off by fires also only damage paint when the action is continued for a long time, as, for example, on bridges below which trains pass at short intervals; under these circumstances the paint is quickly destroyed.

The action of pigments upon the drying oils and boiled oils has been demonstrated. The lead pigments (white lead, red lead) form hard solid masses in combination with the drying oils, which is probably due to the small quantity of oil they require in order to produce a paint of proper consistency. In spite of the hardness and solidity which these paints possess in consequence of the small amount of oil they contain, they exert an extraordinary resistance towards atmospheric influences and water; they produce more durable layers than paints containing other pigments. The pigments require the following quantities of linseed oil or boiled oil to form paint:—

White lead	8–9 per cent.
Red lead	6–8 ,,
Zinc white	22 ,,
Baryta white	7 (?) ,,
Brunswick green	11 ,,
Oil black	27 ,,
Ferric oxide	10 (?) ,,
Burnt Turkey umber	29 ,,
Burnt English umber	20 ,,
Brunswick blue	11 ,,
English ochre	16·5 ,,
Vandyke brown	40 ,,
Raw Sienna	37½ ,,
Burnt Sienna	37½ ,,
Chrome yellow	15 ,,
Levigated chalk	18 ,,

These quantities are, however, not to be regarded as fixed values, very much depends upon the nature of the pigment and its composition; they may be taken as average values. Thus, for example, zinc white requires from 10 to 15 per cent.

and white lead also requires varying quantities of oil. In general, it may be said that pigments of crystalline nature take much less oil than those which are soft and clayey, such as many varieties of ochre. All the pigments of high specific gravity, *i.e.* which take up a small volume, require much less raw or boiled linseed oil than voluminous pigments, so that the volume affords a measure of the oil required. Experience has shown, in the case of oil varnishes, that carriage varnish made with much oil is far more durable than a copal varnish short of oil; thus it almost appears that to the high proportion of oil alone is due this durability. The durability of oil paints exposed to the atmosphere varies in the same manner. We have seen that there are pigments which require little oil, especially white and red lead, whilst zinc white requires more than twice as much. There are similar variations with all other pigments, and it is worth while to consider in what way this difference becomes evident in the paint. When 1 kilo. of a heavy paint is spread out upon a surface, it does not cover so great an extent as 1 kilo. of a light paint, simply because the volume of the heavy paint is smaller than that of the light paint. If the volume of the light paint is twice as great as that of the heavy, the former will cover twice the surface covered by the latter; thus in this case the volume and not the weight is of importance. For example, if 200 grms. of oil be taken to 1 kilo. of dry material, and for a lighter paint 700 grms. of oil to 1 kilo., and if the volume of the latter paint is twice that of the former, there will be 200 grms. of oil in 1 volume of the heavy paint, and 350 grms. of oil in the same volume of the lighter paint. When half the total volume of the lighter paint is spread out over the same surface as 1 volume of the heavy paint, in the first case there are 200 grms. of oil, in the second, 350 grms. of oil, upon equal surfaces. This is only an example, in many cases the differences are considerably smaller, but may be even larger; it was only intended to show here that considerable differences may occur. When a surface of fixed extent is covered with a paint containing a large proportion of oil, the paint will be more durable, for the reasons given, than a coating made with a paint containing a pigment which requires little oil.

Dried layers of oil or paint cannot permanently resist the action of alkaline solutions, caustic or carbonated; they are rapidly attacked on the surface, and finally are completely removed. The resistance towards acids is also relatively small, although it is possible to obtain an uninjured skin of varnish or paint by immersing painted zinc foil in acids, because the zinc is more rapidly dissolved than the skin is attacked.

Spennrath made a number of experiments upon the action of different agents upon paint skins, separated in the above manner, containing linseed oil and graphite (an indifferent pigment), and obtained the following results:—

1. Paint skin, containing linseed oil and graphite, kept for 6 months under *pure rain water.* The skin was still coherent and elastic, but was dull and considerably damaged; it had lost 10·4 per cent. in weight.

2. Paint skin, containing linseed oil and graphite, kept for 6 months under *sea water.* During the experiment fungi developed in the water to a great extent; where they adhered to the skin it was coloured white, otherwise it was uninjured, and had even retained its face. The elasticity was but little less; the loss in weight was 4·52 per cent.

3. Paint skin, containing linseed oil and graphite, kept for 6 months under a *10 per cent. solution of common salt.* The skin was hardly attacked, but was rather dull, the elasticity was unaltered; the loss in weight was 2·4 per cent.

4. Paint skin, containing linseed oil and graphite, kept for 6 months under a *10 per cent. solution of sal ammoniac.* The

skin was externally unaltered, it was shining and elastic; the loss in weight was 3·5 per cent.

5. Paint skin, containing linseed oil and graphite, kept for 6 months under a *10 per cent. solution of magnesium chloride.* The skin appeared to be unaltered, it was shining and elastic; the loss in weight was 1·1 per cent.

6. Paint skin, containing linseed oil and graphite, kept for 6 months under *5 per cent. sulphuric acid.* The skin was unaltered, shining and elastic; the loss in weight was 1·65 per cent.

7. Paint skin, containing linseed oil and graphite, kept for 3 months under *5 per cent. hydrochloric acid* (5 grms. of acid of 1·175 specific gravity and 100 grms. of water). The skin was considerably attacked and was dull; the loss in weight, 12·92 per cent.

8. Paint skin, containing linseed oil and graphite, under *5 per cent. nitric acid.* The skin was destroyed, the remains could not be weighed.

9. Paint skin, containing linseed oil and graphite, hung in a *closed space over acetic acid.* After several weeks the skin liquefied and fell off in drops.

10. Paint skin, containing linseed oil and graphite, hung for 6 months in a *closed space over sea water.* The skin was externally unaltered, was shining and elastic, but somewhat sticky; there was no loss in weight.

11. Paint skin, containing linseed oil and graphite, hung up for 6 months in a *closed space over anhydrous calcium chloride, i.e.* in dry air. The skin was quite unchanged, elastic and shining; the increase in weight was 0·40 per cent.

12. Paint skin, containing linseed oil and graphite, kept in a *closed space over fuming hydrochloric acid of specific gravity 1·175.* In a few days the skin was destroyed and ran down in drops.

13. Paint skin, containing linseed oil and graphite, kept

for 6 months *in an aqueous extract of coal ash* (alkaline reaction). The skin was considerably attacked, had lost in face and elasticity; the loss in weight was 14·8 per cent.

14. Paint skin, containing linseed oil and graphite, kept in a *closed space over nitric acid of specific gravity 1·335.* In several days the skin was destroyed and ran off in drops.

15. Paint skin, containing linseed oil and graphite, kept *under a 1 per cent. solution of soda.* In 3 days the skin began to dissolve, and before many days was completely destroyed.

16. Paint skin, containing linseed oil and graphite, kept in a *closed space over ammonia solution.* The skin was completely destroyed in several days, and ran off in drops.

17. Paint skin, containing linseed oil and graphite, kept in a *closed space over ammonium sulphide.* In a few days the skin was completely destroyed, and ran off in drops.

18. Paint skin, containing linseed oil and graphite, kept in a *closed space over a solution of sulphur dioxide in water.* In a few days the skin was destroyed, and ran off in drops.

19. Paint skin, containing linseed oil and graphite, hung in a *closed space over water at 70° to 80° C. for 24 hours.* The skin was considerably attacked, was dull, and had lost 9·86 per cent. in weight.

20. A paint skin, composed of linseed oil and red lead, was kept for 24 hours in a *closed space in an atmosphere of sulphuretted hydrogen.* It became black, dull, and rugged; the increase in weight was 1·5 per cent.

Spennrath draws the following conclusions from these experiments:—

1. A coat of any paint is destroyed by dilute hydrochloric and nitric acids, gaseous hydrochloric, nitric, sulphurous and acetic acids. Gaseous acids act more rapidly than acids in dilute aqueous solution. Dilute sulphuric acid does not attack paint.

2. Alkaline liquids and gases: ammonia, ammonium

sulphide, and soda solution, rapidly destroy paint. There was no necessity to examine the action of caustic alkalis, which are known to destroy a coat of paint in a few moments.

3. Pure water has much more disintegrating action than solutions of common salt, ammonium chloride, and magnesium chloride, which are known and feared as producing rust on iron. Sea water also has less action than pure water; the salts diminish the action of the water upon the paint. The destructive action of sea water upon oil paints must be ascribed to the mechanical action of the moving water upon the layer of paint.

4. Hot water acts much more rapidly upon a coating of oil paint than water at the ordinary temperature, and destroys it in a relatively shorter time.

5. An aqueous extract of coal ash, in consequence of its alkaline nature, exerts a destructive action upon paint. The fine ash ejected from chimneys is thus a dangerous material when it falls upon paint.

In these experiments various observations led to the conclusion that a high temperature also affects paints in the absence of chemical agents. A consideration of the experiments quoted below leads to the conclusion that higher temperatures (*i.e.* below 100° C., at which decomposition by heat does not occur) are the most subtle and dangerous foe to paint. If a paint skin be exposed to a temperature of about 100° C., in a short time it suffers considerable alteration; it loses its elasticity and pliability, becomes hard and brittle, breaks when bent abruptly, loses weight, and is shortened by several per cent. At temperatures between 100° C. and the ordinary the same alterations occur, but more slowly. At 120° C. a paint skin becomes in a short time as brittle as glass. Spennrath's experiments gave the following results :—

1. A paint skin, composed of linseed oil and graphite, *exposed for 5 days to air at 50° C.*; considerable loss in pliability and elasticity, 4·4 per cent. loss in weight, and 1·2 per cent. shortening.

2. Paint skin, composed of linseed oil and graphite, *exposed for 5 days to air heated in a water bath to 95° C.*; became stiff and brittle, lost 6·07 per cent. in weight, was 3·5 per cent. shorter, and broke when bent sharply.

3. Paint skin, composed of linseed oil and graphite, *exposed for 8 days to a temperature of 95° C.*; broke when bent abruptly, had lost elasticity and pliability; loss in weight 8·5 per cent., shortening 4 per cent.

4. Paint skin, composed of linseed oil and graphite, *exposed for 3 days at a temperature of 120° C.*; was stiff and brittle, loss in weight 9·5 per cent., shortening 5 per cent.

5. Paint skin, composed of linseed oil and red lead, *exposed for 5 days to a temperature of 95° C.*; was stiff and brittle like glass, broke on bending; loss in weight 2·11 per cent., shortening 4 per cent., considerably darker in colour.

6. Paint skin, composed of linseed oil and white lead, *kept at 95° C. for 5 days*; was hard, friable, and brittle, yellow in colour; loss in weight 4·1 per cent., shortening 4·3 per cent.

7. Paint skin, composed of linseed oil and lead sulphate, *kept for 5 days at 95° C.*; was stiff and brittle, yellow in colour; loss in weight 3·9 per cent., shortening 4 per cent.

8. Paint skin, composed of linseed oil and zinc white, *kept for 5 days at 95° C.*; was stiff and brittle, yellow in colour; loss in weight 5·72 per cent., shortening 4 per cent.

9. Skin from paint composed of linseed oil and graphite diluted with 10 per cent. of turpentine, *kept for 5 days at*

95° C.; was stiff and brittle; loss in weight 5·48 per cent., shortening 4 per cent.

10. Skin from paint composed of linseed oil and graphite, with 10 per cent. of drier, *kept for 5 days at 95° C.*; was stiff and brittle; loss in weight 6·4 per cent., shortening 4 per cent.

11. Paint skin composed of poppy oil and graphite, *kept for 5 days at 95° C.*; was stiff and brittle; loss in weight 8·39 per cent., shortening 3·8 per cent.

12. Skin from paint composed of linseed oil and graphite, with 5 per cent. of mineral oil, *kept for 5 days at 95° C.*; was stiff and brittle; loss in weight 6·83 per cent., shortening 4 per cent.

13. Skin from paint composed of linseed oil and graphite, with 10 per cent. of mineral oil, *kept for 5 days at 95° C.*; was stiff and brittle; loss in weight 7·8 per cent., shortening 4 per cent. (The addition of mineral oil was made with the object of ascertaining whether the introduction of an indifferent non-drying oil would cause the paint to retain its original softness and elasticity at a high temperature. The addition of mineral oil is, however, rather harmful than useful. More than 10 per cent. of mineral oil should not be used, otherwise the paint will remain tacky after drying.)

14. Paint skin from "Bessemer paint,"[1] *kept for 5 days at 95° C.*; was stiff and brittle; loss in weight 6·41 per cent., shortening 4 per cent.

15. Skin from paint made from micaceous iron ore, *kept for 5 days at 95° C.*; was stiff and brittle; loss in weight 4·3 per cent., shortening 3·5 per cent.

From these experiments it follows:—All paints lose in weight on heating, and contract; at the same time, they lose their softness and elastic nature, becoming stiff and brittle. Whilst the paint remains on the surface it covers, it cannot contract, the layer of paint is therefore in a state of tension. In proportion to the intensity and duration of the action of heat, in particular when produced by the sun's rays, the tension and also the brittleness of the layer become greater; finally the paint must crack. Thus there arise the cracks which are observed in old paint, especially on iron, and which form a network over the whole surface. When these cracks are formed the surface beneath becomes exposed to the action of rain. The cracks produced by heat are seen very clearly on old painted ceilings. If we consider that the ceiling of a hot room may be at a temperature of 70° C. or more, the appearance of cracks is at once explained.

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[1] A paint for ironwork, composed of ferric oxide and boiled linseed oil.

On comparing the nature of paint skins of different composition, it will be found that those made from graphite appear less stiff and brittle than those made from white lead (? and zinc white), and especially than red lead paint. The last named is so friable and brittle that it at once breaks with the least mechanical action. Now the specific gravity of these pigments is as follows:—

Graphite	2·3
Zinc white	5·42 (?)
White lead	6·43
Red lead	9·07

In an equal weight of paint skin there is thus a far larger quantity of the metallic compounds, and therefore less oil, than in a coat of graphite paint. Thus it is explained why the dried graphite paint is not so hard as coatings of white lead, zinc white, or (especially) red lead; and further, it is clear why red lead paint is valued on account of its hardness. On the other hand, however, zinc white and white lead paints offer much less resistance to the action of heat than graphite paint; thus we conclude that, of two paints exposed to the action of heat, that one will be the most durable which contains the pigment of the smaller specific gravity.

White paints made from zinc white, white lead, and lead sulphate become very yellow on heating, red lead paint turns dark; indeed, in general, we may say that a pale coloured paint becomes yellow and brown when gently heated, and that a tolerably dark paint becomes darker. This change is due to the oil turning yellow or brown on heating. For the same reason ceilings, painted with pale oil paints, darken and finally become quite brown in places over gas or petroleum lamps. The darkening is generally ascribed to soot given off from the flames. As a matter of fact, the change is due to heat, which is proved by the fact that a similar change occurs above stoves. The Welsbach incandescent gas light also browns painted ceilings, although it gives off no soot. The hopes which had been based upon the use of the electric light were in this respect not fulfilled. Spennrath has observed that painted surfaces in the neighbourhood of incandescent electric lights were browned, if the latter were not at too great a distance from the paint.

CHAPTER VII

BOILED OIL SUBSTITUTES

In view of the ever-increasing competition and the ever-decreasing margin of profit in the manufacture of boiled oil, and of the paints made from it, it will readily be understood that endeavours have been made for some time to produce substitutes for boiled oil, which are either brought into the market, under that description, for use in place of boiled oil in preparing paints, or are employed to cheapen boiled oil so that it can be sold under the market price—an unfortunate use. When a boiled oil substitute is sold under that name nothing can be said against the transaction, and the purchaser will know that the material cannot completely replace boiled linseed oil. But if boiled oil mixed with a substitute is sold, and the consumer is led to believe that it is only through exceptionally favourably circumstances that "good" boiled oil can be sold at so low a price, or if boiled oil mixed with a substitute is delivered as pure and unadulterated, the transaction is at the least doubtful, to avoid a more severe but more appropriate term.

Attempts to prepare a substitute for boiled linseed oil date back over a considerable period; the use of other fatty oils, non-drying, is generally excluded, if not at all times, since as a rule other oils cost more than linseed oil, and since, finally, a boiled oil substitute is required to dry. Thus, supposing that non-drying oils were cheaper than the cheapest of the drying oils (linseed oil) for a lengthy period, only very small quantities of the non-drying oils could be added, and the difference in cost would not be large enough. The other drying oils—poppy, walnut, candle-nut, and even Chinese wood oil, which last might replace linseed oil with particular advantage—are all dearer than linseed oil, which is used to adulterate the other oils. Attempts to make cottonseed and earthnut oils dry, have indeed led to favourable results, but these oils, generally used as edible oils, are higher in price than linseed oil, if the latter has not reached an exceptionally high figure. Thus all the materials, which are not very different in value from linseed oil, are *à priori* excluded from use as boiled oil substitutes.

Rosin is one of the next most suitable materials for the preparation of boiled oil substitutes, and in fact it is frequently introduced into boiled oil; it is simply melted over the fire, and added to the boiled linseed oil. But in this case, also, small quantities could exert no considerable influence upon the cost, and with larger quantities the boiled oil would be so thick that it would be too far removed from the requisite degree of fluidity. Rosin also possesses the great disadvantage that it forms a thick, sticky deposit in boiled oil, in which it can readily be recognised; in addition, lead and zinc pigments, which are the most important, thicken with boiled oil containing rosin, and even separate completely under certain conditions as lead and zinc resinates. Such material is useless, quite apart from the fact that coatings of boiled oil containing rosin never dry completely, but remain tacky. Since now a boiled oil substitute must produce a coating which dries hard, the simple introduction of rosin into boiled oil is not sufficient to yield an adequate substitute.

The next most likely material is rosin oil, thin or thick. It has been used for many years as a boiled oil substitute, yet it has many disadvantages, not the least of which is that it separates after the lapse of a longer or shorter time, especially in winter. Recently greater attention has again been paid to rosin oil, especially since a means has been found, in the resinates of lead and manganese, to make it dry to a certain extent. The experiments made by Weger with rosin oil and rosin oil mixtures, in which the drying and oxygen absorptions were determined, are very interesting. The following are the results:—

1. *Refined rosin oil*, in which 6 per cent. of lead-manganese resinate had been dissolved at 150° C.—

Weight of oil . .	0·0379 grm.	
Increase after 18 hours .	0·0013 „	= 3·4 per cent. (still very tacky).
„ 42 „ .	0·0058 „	= 15·3 per cent. (dry).
„ 3 days .	0·0067 „	= 17·6 „
„ 5 „ .	0·0073 „	= 19·3 „
„ 9 „ .	0·0075 „	= 19·8 „

2. *Boiled rosin oil*, consisting of 80 parts of refined rosin oil, 20 parts of linseed oil, and 6 per cent. of lead-manganese resinate, prepared in the cold—

Weight of oil . .	0·0442 grm.	
Increase after 22 hours .	0·0039 „	= 8·8 per cent. (nearly dry).
„ 2 days .	0·0065 „	= 14·7 per cent. (dry).
„ 3 „ .	0·0077 „	= 17·4 „
„ 5 „ .	0·0077 „	= 17·4 „
„ 9 „ .	0·0081 „	= 18·3 „

3. *Boiled rosin oil*, consisting of 100 parts of refined rosin oil, 10 parts of linseed oil, and 5 parts of lead-manganese resinate—

Weight of oil . .	0·0388 grm.	
Increase after 16 hours .	0·0020 „	= 5·2 per cent.
„ 24 „ .	0·0034 „	= 8·8 „ (almost dry).

Increase after	3 days .	0·0054 grm.	= 13·9 per cent. (dry).
„	11 „ .	0·0066 „	= 17·0 „
„	26 „ .	0·0076 „	= 19·5 „
„	93 „ .	0·0069 „	= 17·8 „

But here also the same inconveniences arise as with rosin. The mixtures with boiled linseed oil do not dry completely, the coatings remain tacky; the mixtures are very liable to decomposition on keeping, and the lead and zinc paints made from them become hard, so that they cannot be used. Recently, boiled oil substitutes have been made from rosin oil which turn out to be usable. Also with fish oil favourable results are said to have been obtained, though this may legitimately be doubted, considering the nature of fish oils.

Mineral oils have been used repeatedly to reduce boiled oil, the latter being thickened by air and mixed with benzine. The mixtures are said to be found suitable. The ordinary thick or thin mineral oils, cylinder oils, spindle oils, paraffin oils, and debloomed oils may be mixed in small quantity with boiled oil; they do not then particularly retard the drying, as is mentioned in another place. Large additions, on the other hand, as the author observed many years ago, separate in drying from the boiled linseed oil, and, when the latter has become a solid layer, remain still wet upon the dry skin, and may readily be wiped off.

No boiled oil substitutes, whatever their advantages, are in a position to replace boiled linseed oil completely; they will always produce more or less friable skins, readily rubbed off; they will not withstand atmospheric influences, and will (with a single exception) be wanting in the desired elasticity, which is the principal characteristic of boiled linseed or other drying oil. It is exactly the same with rosin in oil varnishes; none of the artificial products—rosin soaps, esters, copal substitutes, etc.—can ever replace the copals; they are always inefficient substitutes.

Substitute for boiled oil from rosin soaps.—The acid of rosin (sylvic acid) forms solid compounds with lead and manganese, used as driers, in which the sticky and soft nature of rosin is more or less absent, and which may be dissolved readily and almost completely in one or more of the ordinary solvents: benzene, petroleum ether, solvent naphtha, turpentine, petroleum, etc. These rosin soap solutions produce coatings, which are hard and do not soften or stick, and well resist the action of water. The fatty acid compounds of the heavy metals also possess these valuable properties to a greater or less extent; generally they are not as applicable as the rosin compounds, on account of the higher market price of fats (lard, cocoa-nut oil, palm oil, etc.), so that as a rule they cannot be used.

If ground rosin be dissolved in boiling soda solution, an operation which requires little time, and the solution be precipitated hot or cold by the aqueous solution of a metallic salt, *e.g.* aluminium sulphate, ferrous sulphate, zinc sulphate, copper sulphate, etc., generally speaking, by metallic salts, the acids of which can drive out the carbonic acid from the soda-rosin solution, the metallic sylvates are precipitated as coherent masses, more or less sticky whilst hot, and white, brown, green, etc., in colour, according to the nature of the metal. If cold solutions are used in the precipitation, the compound forms small particles, which do not unite until heat is applied. Of the metallic sylvates, the zinc and iron compounds are especially adapted to the preparation of boiled oil substitutes, and in less degree the aluminium and copper compounds. The iron compound is the most suitable; it is extremely hard, remains elastic even after years, is never sticky, and is readily and completely soluble in solvents. Unfortunately, it possesses an intense rust-brown colour, which it imparts to pigments with which it is mixed, so that it can only be used for dark paints: Indian red, ochre, brown, and black. Zinc sylvate has been found to be the most suitable for the preparation of a really practical substitute, which dries well, is not sticky, and may be ground with white lead and zinc white without producing hardening or decomposition. From it a substitute for boiled oil can be made, which completely satisfies all requirements, and may be mixed with boiled oil in any proportions. This substance is prepared as follows. The apparatus required consists of a large iron pan with an outlet and a tap half-way up, and a smaller iron pan, both of which may be heated by fire or by steam.

Quantities.

15	kilos. of	ordinary soda crystals.
25	„	rosin, pale or dark, according to the colour required.
15	„	zinc sulphate.
250	„	water.
50	„	water to dissolve the zinc sulphate.
12·5	„	boiled oil.
25	„	petroleum or petroleum ether.

Process.—250 kilos. of water are heated to boiling in the large pan, in it 15 kilos. of soda are dissolved, and then, the liquid being kept boiling, 25 kilos. of rosin in fine powder (ground in a mill) are introduced gradually, a fresh quantity being added only when the last has dissolved. The pan in which this operation is performed must be large enough to contain double the above quantities, that is, about 600 kilos. When all the rosin has dissolved the zinc sulphate solution, made in the second pan from 15 kilos. of zinc sulphate and 50 kilos. of water, is gradually poured into the soda-rosin solution whilst stirring. The mixture effervesces and rises, so that the precipitation must be cautiously conducted. When all the zinc sulphate solution has been added and the zinc sylvate has separated, the mixture is again heated to boiling. When the temperature is sufficiently high the zinc soap collects to a resinous mass; after cooling, the water, which contains Glauber's salt, is drawn off, and thrown away or evaporated.

The zinc sylvate may now be treated in two ways:—

(1) The mass is taken out of the pan, dried in thin layers upon frames in a drying room, and then dissolved in the heated boiled oil.

(2) The mass is melted with boiled oil whilst still wet, in which operation care is necessary, since the water may cause the mixture to overflow.

In either case the heating is continued at a temperature of 120° to 130° C. until a portion of the melted mass, dropped upon glass, is seen to be quite clear, transparent, and without turbidity. When this is the case the pan is removed from the fire (or if the pan is built in, the fire is extinguished), the contents are allowed to cool to 50° to 60° C., and then, whilst stirring, 25 kilos. of high-boiling petroleum or 25 kilos. of petroleum ether are added, according to the cost and the consistency desired. The boiled oil substitute made from petroleum ether naturally dries in considerably shorter time than that made from petroleum. The finished product is tanked, or, if it is to be sent away at once, is filtered, which is best done in a bag-filter.

Different consistencies of the boiled oil substitute are obtained by using larger or smaller quantities of petroleum or benzine, which is merely a solvent; the quantity of boiled linseed oil may also be reduced to 5 kilos. for 25 kilos. of rosin; in this case the cost is lower.

In case the preparation of zinc sylvate offers difficulties on account of the effervescence and the large pan required, the following method may be adopted. The soda-rosin solution is poured into a large wooden tub, the zinc sulphate solution added, and the mixture thoroughly stirred, after which a solution of 100 grms. of caustic soda is introduced. When the zinc soap is completely precipitated the water is run off, and the mass transferred to a large sack (an ordinary sack such as is used for chalk, fruit, etc.), which is suspended from several points. The liquid begins to run away when the bag is filled, and the zinc soap remains behind. When the sack is nearly full, it is tied up, the liquid removed by loading with weights, and the bag with its contents brought into an ordinary spindle press, in which the press plates are screwed together until the liquid is almost completely expressed, so that the cake is nearly solid. It is taken out of the sack, broken up and completely dried in thin layers in the air or in a drying stove; it is then utilised according to the previous method.

Oster's linseed oil substitute.—5·23 parts by weight of pale Burgundy pitch are melted and mixed with 2·735 parts of crude cottonseed oil and 0·5 part of thick Dutch stand oil, both previously heated to 80° C. Then 3·5 parts of petroleum at the same temperature are added, and the whole heated; after cooling, 0·025 part of oil of valerian and 0·025 part of essence of mirbane are introduced, and the solution allowed to brighten. It is stated that this oil may be used instead of linseed oil in all its applications. The drying power may be increased by previously heating the cottonseed oil with 3 per cent. of litharge.

Goldblum's boiled oil substitute.—This product is not only considerably cheaper than linseed oil, the advantages of which it possesses in increased measure without having its unpleasant smell and danger of causing fire; it has considerably greater drying power, and may be mixed in all proportions with paints of every kind.

This substance is made by dissolving ground rosin in a suitable quantity of benzene, benzine, or naphtha whilst stirring; for example, 50 parts by weight of rosin in 100 parts by weight of solvent. The solution is then treated under constant stirring with coarsely powdered soda or another alkaline carbonate, which, without uniting with the rosin itself, by an action partly mechanical and partly chemical causes the impurities to separate as a precipitate. At the same time, the water which occurs in ordinary commercial rosin is removed by the calcined soda. The residue and the excess of soda are then separated by filtering from the liquid, which now forms a clear solution, and does not become turbid on long standing.

The alkaline carbonate may be used in the proportion of 12 to 15 parts to 50 parts of rosin. Other alkaline carbonates may be used instead of soda, which cannot, however, be replaced by the carbonates or oxides of the alkaline earths, since the latter form from the rosin metallic soaps soluble in benzine, which would separate and make turbid the finished product. In this process, on the contrary, the rosin itself is in no way attacked, and the applicability of the product is not only not diminished but considerably increased. In comparison with the preparations obtained by treating rosin with lime or other alkaline earths, the products of this process have the advantage that they do not possess the stickiness caused by the lime soap.

Pietzker's boiled oil substitute.—Ordinary rosin is melted, 50 per cent. of manganese resinate or linoleate or the corresponding lead or copper compound is dissolved in it, and the mixture heated to boiling. Then 20 to 40 per cent. of the following compounds is dissolved in water, of which an equal quantity is taken—potassium, sodium, calcium, magnesium, lead, zinc hyposulphite; potassium, sodium, calcium, barium, strontium, magnesium, lead, manganese, zinc sulphite; potassium, sodium, barium, strontium, calcium sulphide. When these compounds are not soluble in water they are stirred with the same quantity. In the meantime the rosin has been allowed to cool to 100° C., the solution is now added to it, and the mixture well stirred, when a soft, turbid, yellowish-white mass is produced. This is then heated at 100° C. until all the water is driven off and the mass is again quite clear. The sulphur compound falls to the bottom; 5 to 20 per cent. of the rosin so treated is dissolved in heated rosin oil in which has been dissolved 3 to 5 per cent. of the driers mentioned at the commencement. The same result may also be obtained in the following manner:—5 to 15 per cent. of rosin and 5 per cent. of one of the above driers are dissolved in rosin oil. Then 5 per cent. of the sulphur compound is dissolved in 3 times as much water, the solution mixed with the oil, so that an emulsion forms if possible, which is then heated at 60° to 80° C., according to the quantity, for ½ to 2 hours with constant stirring. The oil is allowed to stand at the same temperature to separate and brighten.

Pietzker's boiled rosin oil.—One of the driers before mentioned is dissolved in rosin oil, 3 to 5 per cent. of resinate or linoleate of manganese or lead is sufficient. Then the rosin oil is mixed at 50° C., to which it has been heated, by means of a stirrer with a strong solution of the following salts (the exact strength of the solution is unimportant): the chlorates, chlorites, hypochlorites, and chlorides of potassium, sodium, ammonium, barium, strontium, and calcium. The mixture is allowed to stand at the same temperature until the oil and solution have separated, and the former has become clear.

Boiled rosin oil.—(A method tested in practice.) For this boiled oil substitute either crude thick rosin oil or thin refined rosin oil may be used. The essential part of the process is that it is absolutely necessary to treat the rosin oil with precipitated (not fused) manganese resinate at 130° to 140° C. for at least 3 to 4 hours, so that the drier may be dissolved completely. The proportions are—

(1)	200	kilos. of	crude rosin oil.
	15	„	precipitated manganese resinate.
	20	„	linseed oil.
	100	„	boiled linseed oil.

(2)	100	kilos. of	refined rosin oil.
	5	„	manganese resinate.
	10	„	boiled linseed oil.

The raw or boiled linseed oil is heated with the manganese resinate until the latter has dissolved to a dark reddish-brown solution, which action generally takes place at 150° to 160° C., the crude or refined rosin oil is next slowly added whilst stirring; the mixture is then heated at 130° to 140° C. for 3 to 4 hours. After this time the boiled oil of formula (1) is added, and the mixture heated for 30 minutes more at the same temperature; the finished product is then left at rest. The proportion of boiled linseed oil may be altered as desired, more or less may be used; boiled linseed oil may also be added to the oil made from refined rosin oil, by which the quality is improved and the cost proportionately increased. Boiled rosin oil made in this way dries well; it does not decompose either on long keeping or in combination with pigments (in particular there is no action with white lead or zinc white); it answers to all the requirements which may legitimately be made of a boiled oil substitute.

Boiled rosin oil—Halphen's method.—1. *With manganese resinate or oleate.*—60 parts of manganese resinate or oleate are dissolved in ordinary rosin and the mixture heated until it is quite clear. 20 to 40 parts of a sulphite or hyposulphite are dissolved in an equal quantity of water. The two liquids are mixed when the rosin solution has cooled to 100° C. A yellowish-white turbid mass of the consistency of tar results; it is heated at 100° C. until all the water has evaporated, when it is clear. 5 to 20 parts of this mass are mixed with 100 parts of rosin oil to which 3 to 5 parts of manganese resinate or oleate have been added.

2. *With lead and manganese oxides.*—100 parts of rosin oil, 3 parts of litharge, and 10 parts of manganese peroxide are boiled in a tinned pan for 2 hours without stirring, and then allowed to settle during 24 hours. A pale oil is obtained, which dries better than ordinary linseed oil.

Boiled oil substitute from fish oil.—For several years a number of painters have replaced linseed oil by a product obtained from fish oil, which is stated to have better qualities in conjunction with a lower price. Into a wooden barrel are brought 144 litres of good vinegar, 6 kilos. of litharge, and 6 kilos. of zinc sulphate; the barrel is rolled about for a long time, and the liquid then poured into 100 litres of fish oil—that obtained in the Pacific is the best. The mixture is well stirred, and then left at rest for 24 hours; when the clear oil is drawn off, $\frac{7}{8}$ of the original quantity is obtained; 54 litres of linseed oil and 9 litres of turpentine are at once added. The liquid is left at rest for several days, and then drawn off. The residue is mixed with an equal volume of milk of lime, and used for painting wood and iron which are exposed to the air.

Sommer's process.—100 parts by weight of refined fish oil are dissolved in 75 to 100 parts of a solvent such as petroleum, benzine, etc., and a suitable quantity of chloride of sulphur then added. The acids, which are formed, are removed by sending a current of dry air through the mixture, which takes with it the hydrochloric acid. The remaining acid is absorbed by 2 to 3 parts by weight of turpentine, finally a small quantity of manganese soap dissolved in linseed oil is added.

Boiled oil substitute from Grisée oil.—The raw material is obtained from a well bored at Grisée in Java; it has a syrupy consistency, is brown in colour, forms emulsions with salt water, and has the specific gravity 0·97. The oil is refined by heating to about 80° C. Occasionally the emulsion is previously treated with salts or light solvents, such as benzine, in order to facilitate the partial separation of the water. In order to obtain oil drying within a definite time, the following treatment is adopted:—The oil after mechanical purification is partially distilled, the thick residue is then dissolved in volatile solvents, such as benzine, chloroform, carbon tetrachloride, etc.; the time required for drying is determined by the solvent chosen. The distillation of the oil is carried out at 300° C.; the varnish-like residue then constitutes 40 per cent. of the original oil.

Boiled oil substitute from the residues of petroleum refining.—The endeavours to prepare a boiled oil substitute from the tar obtained in treating crude petroleum with sulphuric acid are not new; in 1863 Adolph Michigan took out an American patent, according to which the acids produced in refining are treated with 40 per cent. of water and 20 per cent. of caustic alkali; the resulting fluid tar was said to be used for mixing with paints. The patents of Zemy followed in 1876 and 1877; the acid tar was neutralised and washed with water and alkalis, and then oxidised by means of a current of hot air, or the readily volatile constituents were removed by heating in pans with strong soda solution of 20° B. The addition of substances to accelerate the oxidation—lead and manganese oxides—was also recommended. When the mass was quite hard on cooling, the operation was finished; it then formed dark brown solutions on treating with organic solvents.

R. Zalozieki has recently worked out a process of practical value for preparing a drying oil. The raw material is obtained by treating oils from various sources with strong sulphuric acid, allowing to stand, and drawing off the acid tar, which is then stirred with ½ to 1 part of water by weight. Three layers form, the lower, containing dilute sulphuric acid, deep reddish-brown in colour; the two upper layers contain the precipitated products of the action of sulphuric acid upon petroleum, consisting of polymerised constituents of the oil, organic sulphonic acids, and sulphones. From these two upper layers a vehicle for pigments is manufactured; they are drawn off, heated in pans with an equal volume of water, and neutralised, whilst stirring, by caustic alkalis or basic oxides (lime, baryta, magnesia, etc.), or by carbonates and organic bases. The essential product is then obtained as a red homogeneous mass, which only requires to be dissolved in the proper solvent. For this purpose a mixture of alcohols and hydrocarbons is used; it is obtained by mixing amyl, methyl, and ethyl alcohol with benzine, turpentine, or light tar-oil. The mass obtained as stated above is made anhydrous by drying or treating with superheated steam, and is then introduced in the cold into the above solvent. It dissolves almost completely, the uniform solution is then drawn off. Two different solutions may also be obtained by stirring the substance, without further treatment, into a mixture of equal parts of 50 per cent. spirit, benzine, and turpentine in the cold; on standing, two layers are obtained, the upper containing oil and hydrocarbons, the lower, alkalis; the layers are filtered and separated. The final product is theoretically a solution of metallic soaps. The product is fairly stable, does not change when exposed to air and light, but is acted upon by water. Fresh acid tar only is suitable for preparing this substance; in it the polymerised constituents completely predominate over the sulphonic acids, which are harmful in large quantity, and cause the product to be readily acted upon by water. On the other hand, the petroleum acids are very valuable for this purpose, since they produce the necessary driers. In previous researches these acids were neutralised and thus made inactive, which was the cause of the failures in treating the acid tar.

CHAPTER VIII

THE MANUFACTURE OF SOLID AND LIQUID DRIERS FROM LINSEED OIL AND ROSIN

LIQUID driers have been made for many years from linseed oil, and used in large quantities in practice, mixed with paints and varnishes, in order to accelerate drying. Originally, and until the last 25 years, they were called siccatives, and every specialist, painter, and varnisher knew that by this term a solution of linseed oil lead (or manganese) soap in turpentine was understood. In addition to this siccative, which was made in colours varying from pale yellow to dark brown, a viscous substance, made in a similar manner, was known as "boiled oil extract"; it was intended to make linseed oil dry, simply by mixing the two substances without raising the temperature. Since that time the use of the word siccative has quite changed; it is now used to designate those substances mentioned previously, generally metallic oxides and their compounds, which are added to linseed oil or other drying oils, at higher or lower temperatures, in order to make them capable of absorbing oxygen from the air and of drying by its action. The term is not well chosen, having regard to what is understood in practice as a siccative, and has been replaced in every case in this book by the term "drier." The word siccative is derived from *secco*, that is, dry; it should be restricted to its original meaning, the general use of the term siccative for both solid and liquid driers leads to many misunderstandings. [In the following pages the more usual

English name—terebine—is applied to the liquid driers or siccatives.]

In recent years the driers introduced under the terms: siccative, terebine, oleobenthine, xerotine, cementine, rapidine, and other fantastic names more or less happily chosen, have been composed according to the following methods:—

1. Solutions in turpentine of lead and manganese linseed oil plasters, differing widely in composition and mode of manufacture.
2. Solutions in turpentine or benzine of lead resinate and manganese compounds.
3. Solutions in drying oils, generally linseed oil, of lead resinate and manganese compounds.
4. Solutions in turpentine of so-called hard resins, or even of rosin.

Of these, only the first three can really be regarded as driers; solutions of hard resins and rosin can impart no drying power, and should not be sold by reputable makers.

Terebines vary greatly in colour and consistency,—in colour, from pale yellow to dark brown; in consistency the variation is not so great, it lies between that of a liquid almost as mobile as turpentine and that of varnish. In former years consumers demanded thick terebines, which, however, were generally not clear, and left much to be desired in drying capacity. Now only thin terebines are made, which contain the linseed oil lead or manganese plaster completely dissolved. Terebine should possess the following properties:—

1. *The colour* should be as pale as possible, so that white paints are not tinged and given a brownish hue. The colour of the terebine is unimportant in the case of dark paints, and it should be remarked that dark varieties, as a rule, possess far greater drying powers than pale.

2. *The consistency* should lie within the limits mentioned above, yet it may easily occur that a thin terebine has far

greater drying power than a thick preparation, since this property depends upon the complete solution of the lead and manganese compounds.

3. *Clearness.*—Every terebine, pale or dark, should be clear; turbidity is due to undissolved or separated compounds, which possess no drying power.

4. *Drying power.*—This is the most important property of a terebine; it varies in an extraordinary degree according to the composition and the care with which the manufacture is conducted. The opinion as to the manner in which this property is to be tested varies to a similar extent. The terebine is required to dry in thin layers upon metal or glass within 15 minutes, others extend the time to an hour, others again determine the time required to dry when the terebine is mixed with ochre to the consistency of cement, *i.e.* to become so hard that the mass can be separated from the surface beneath, and so on. All these tests give no correct estimate of the drying power, for a terebine which does not answer to them may possess remarkable drying power, and, on the other hand, a preparation which completely answers these requirements may turn out to be a bad drier, which would be the case with solutions of the hard resins. The only safe method for testing the activity of a terebine is to mix it in a definite proportion with tanked or filtered linseed oil, to spread out the mixture upon metal or glass in a thin layer after the turbidity has settled, and to observe the time the oil requires to dry. In general it would be required of a good terebine that linseed oil, when mixed with 5 per cent., should dry within 24 hours; if the oil dries in a shorter time and with less terebine, the preparation is of better quality, but if more than 24 hours are required, or larger quantities of the terebine, the quality is to be regarded as unsatisfactory.

Terebines, as we have seen above, may be divided into three classes; the preparation of those belonging to the first class, made from lead or manganese plaster, will alone be given in detail. In regard to the two other classes, we shall merely give a method for preparing the solid compounds, since the method for obtaining terebine from these only requires a simple process of solution with the application of heat.

Terebine from linseed oil and lead and manganese compounds.—As we have already seen, terebines are solutions of linseed oil lead or manganese plasters in turpentine. In the first place, we must consider the correct and appropriate method of obtaining the intermediate product. The principle of the process is very simple: linseed oil is boiled with substances containing the correct quantities of lead or manganese oxides for so long and at such a temperature that a plaster results, *i.e.* until the linseed oil has reacted with the metallic compounds to form a solid friable mass, slimy in places, which dissolves completely in solvents, especially in turpentine, producing a solution which in clearing forms very little deposit, which latter should contain no metal. The quantity of this deposit depends upon the lead and manganese compounds employed; if these contain valueless substances in addition to the active constituent, the former will make the oil thick but will not act upon it, so that a larger deposit will be formed than when substances are used which contain only oxides or their compounds.

The substances which are principally used in the preparation of terebines are—

Lead compounds.—Litharge, red lead, white lead, lead acetate;

Manganese compounds.—Manganous borate, manganic hydrate, manganese resinate, pyrolusite.

Formerly there were also used: low qualities of white lead (containing chalk or barytes), umber, and shale brown (containing manganese). These substances are no longer used, since they make the oil unnecessarily thick without producing a proper action, and since the finished product brightens with difficulty. Even pyrolusite has almost gone out of use, because manganic hydrate, on account of its purity, gives considerably better results. All the substances mentioned must be as free from water as possible; if it can be arranged, they should be exposed in thin layers for several days at a temperature of 25° to 30° C., so that any moisture may be given off. Sugar of lead, which contains large quantities of water of crystallisation, is treated in a special manner in order to remove this water. It is melted in an enamelled iron pan over a moderate fire, and heated at about 120° C., with constant stirring, until the water is almost entirely driven off and the liquid begins to produce a powder. It is then poured out upon plates, allowed to cool, powdered, and preserved in closed vessels. It is important not to raise the temperature far above 120° C., otherwise the acetic acid will decompose, which is not the intention. If ordinary crystallised lead acetate be used, as is even now frequently the case, decomposition occurs in the finished product on account of the action of water, and the whole batch is made unsaleable. The cause of this decomposition is unknown to many manufacturers, since they are not acquainted with the materials and principles of the process.

The quantities of the lead and manganese compounds, which are used in preparing terebine, vary to an extraordinary degree. As a rule, the driers, as they may briefly be designated, are combined, *i.e.* a certain quantity of the two lead oxides is used together with a definite but small amount of the manganese compound. The reason for the use of different quantities of driers is that the same temperature is not always employed. If a smaller quantity of driers be used, suitable plaster is obtained in a short time at high temperatures, at which the reaction takes place rapidly; if larger quantities of driers be used at a lower temperature, a longer time is required to effect their combination with the linseed oil. Very divergent opinions are held as to which is the better method; the author's experience has taught him that smaller quantities of driers, a high temperature, and consequently a rapid reaction, produce a much better product than long boiling. In this way he obtains a terebine which clears quickly, dries well, and communicates good drying powers to oil. Terebines made at a low temperature clear slowly; they dry well, but communicate less drying power to oil. The last mentioned is the most important property of terebine. The quantity of the lead and manganese compounds used in the preparation of terebine should be 70 to 90 per cent. of the weight of the linseed oil.

The vessels for the manufacture may be chosen as desired; the fuel may be charcoal, coal, or gas, according to local conditions. Steam can be used only when superheated; steam at ordinary pressures would not produce the requisite temperature, and too long boiling would be necessary. Small pans, capable of being moved by two men, are more advantageous, since the boiling mass frequently froths up, when the pan must be removed from the fire. The cooling of large pans built in brickwork is difficult to accomplish, on account of the heat retained by the setting. Whatever form of pan is used, its capacity must be at least six times the volume of the linseed oil and driers to be heated in it.

In preparing the liquid driers obtained from linseed oil and metallic oxides, the solvent naturally plays an important part, and it will not be superfluous to make a few remarks in this connection in order to make clear the points which must be considered. Linseed oil lead plaster, the essential constituent of all terebines which are not simple resin solutions, is, according to the method of preparation, either sticky or friable with a certain degree of softness; it requires a long time to become really hard. In order to convert this lead plaster into a liquid drier, it must be diluted with a solvent whilst it is still liquid, which is the usual method, or it must be dissolved later on with the application of heat. The solvents used for this purpose are turpentine of various sources, petroleum distillates (benzine) of various densities and volatilities, or coal-tar distillates—benzene, toluene, solvent naphtha, etc. In consideration of the large quantity of this solvent employed, which frequently amounts to 4 to 6 times the quantity of linseed oil used for the plaster, it is of interest to know whether it plays any part in the drying process or merely volatilises. In regard to (spirits of) turpentine, which is used as a solvent almost exclusively at the present time, in spite of its high price, the action of light and air must be considered. It should be stated, however, that the above-mentioned petroleum and coal-tar distillates are likely to restrict the use of turpentine on account of their low price. Turpentine adulterated with these distillates and turpentine substitutes, composed essentially of them, are at the present time important articles of commerce.

Turpentine (spirits of) rapidly changes on standing in the air in open vessels, especially in the presence of light; it becomes thick, the specific gravity and boiling point rise, the solubility in 90 per cent. alcohol increases; at first neutral, it becomes acid; it resinifies and, as the technical expression runs, turns rancid. Formerly such turpentine was termed ozonised, since it has strong oxidising properties. All these changes are due to slow oxidation by the oxygen of the air. Normal American turpentine, 0·867 specific gravity, after standing for 7 weeks in a closed bottle partially filled with air, had the specific gravity 0·897, and gave a clear solution with 3 to 5 volumes of 90 per cent. alcohol, whilst the original substance required 6 volumes. Another sample of American turpentine had, after long standing, the specific gravity 0·913, and gave a clear solution with 3 volumes of 90 per cent. alcohol. A sample of French turpentine, after keeping for 4 years in a tightly corked bottle, not quite filled, altered in the following manner:—

	Original normal Turpentine.	After four Years.
Specific gravity	0·871	1·009
Optical rotation	– 29° 55′	– 19° 18′
Volume of 80 per cent. alcohol required to form a clear solution	20	1

The oxidised oil was soluble in every proportion in 90 per cent. alcohol.

The varieties of turpentine absorb oxygen in different proportions, as is seen from the following table:—

Variety of Turpentine.	Oxygen Absorption. Volumes.
French	100
Russian	100
Swedish	100
Swiss	89·4
American	78·9
Swedish, adulterated	52·6
American	42·1
Eucalyptus oil	75·0

Thus 1 c.c. of French spirits of turpentine, which is recognised to be the best, can absorb 100 c.c. of oxygen.

Schönbein supposed that turpentine became charged with ozone during the oxidation, the oxygen of the air being changed by the turpentine into the acid modification. More recently, Kingzett, Bardsky, and Papasogli have shown that oxidised turpentine contains no ozone, but hydrogen peroxide. Loew showed that turpentine, oxidised in the presence of moisture, contains other substances in addition to hydrogen peroxide, for it separates iodine from potassium iodide, which is not a property of hydrogen peroxide. This action is rather to be ascribed, as Kingzett had already assumed, to organic peroxides, which decompose with water in such a manner that hydrogen peroxide is finally formed, apparently with the intermediate production of hydrated peroxides. The explanation of these processes has been given recently by Engler and Weissberg, who showed that when absolutely dry turpentine is made "active," neither hydrogen peroxide nor ozone is formed. They also showed that turpentine makes oxygen "active" most rapidly at 100° C., and that above 100° C. no "active" oxygen is formed, but instead the "active" oxygen is used to oxidise the turpentine.

Turpentine, charged with oxygen in the presence or absence of moisture, is able to transfer the oxygen to substances not directly oxidisable by the oxygen of the air, thus, as has been stated above, iodine is separated from potassium iodide, also indigo solution is bleached, and arsenious acid oxidised to arsenic acid. "Active" turpentine retains its properties for years when kept in the dark.

The products of the oxidation of turpentine are not well known. It has been shown with certainty that formic, acetic, and camphoric acids are formed. A small quantity of an aldehyde, corresponding in composition to camphoric aldehyde, has been found; it possesses an overpowering odour, and is apparently the cause of the similar odour of "rancid" turpentine. These oxidations and alterations of turpentine take place more rapidly when heated air saturated with water vapour is passed through.

By the action of direct sunlight upon moist turpentine in the presence of air, or better, of oxygen, pinol hydrate is formed, which crystallises in leaflets or needles, according to the solvent; its inactive modification melts at 131° C.

By these scientifically ascertained facts it is clearly shown that turpentine is in a high degree capable of absorbing oxygen and again giving it up, as in the drying of varnishes, terebines, and paint, thus acting as an oxygen carrier. The action of turpentine as an oxygen carrier is very clearly shown by the case detailed below. In the previous year a manufacturer of oilcloth appealed to the author for an explanation of the following phenomonen :—A marble oilcloth, varnished by hand, was white; whilst, when the same varnish was applied with the machine, the yellow shade which appeared on application was not lost, but the cloth remained yellow. There was no difficulty in explaining the cause, since it was known that in varnishing by hand the varnish was diluted with turpentine, but in varnishing with the machine, with benzine. This explanation was not given at once, since it would have been received with doubt by the experienced manufacturer and by his foreman of 35 years' standing. The author had samples made by hand and by machine, the varnish being diluted in each case with turpentine. The coats of varnish made with turpentine became colourless, whilst the varnish diluted with benzine remained as yellow when dry as were all the samples immediately after application. Thus only the absorption of oxygen by the turpentine could have caused the bleaching of the yellow varnish, and there is no doubt that this oxygen absorption has a considerable influence upon the drying and hardening of varnishes and terebines, and that account must be taken of this influence. Ten years before, when the first turpentine substitutes were introduced, the author showed that varnishes made from them dried far more slowly than those made with turpentine, the varnishes being otherwise quite the same.

The substitutes for turpentine, which generally consist of benzine, rosin spirit, solvent naphtha, camphor oil, and larger or smaller quantities of turpentine, or even of these hydrocarbons mixed with traces of an essential oil to improve the smell, may be similar in specific gravity to turpentine, but an absorption of oxygen can never occur; these substitutes therefore considerably decrease the drying power. In this connection Lippert remarks as follows:—Although the substitutes for turpentine—fractions of petroleum and benzine—are objectionable in the first place on account of their smell, yet it would be profitable to redistil them, considering the present high price of turpentine. By redistillation the smell would be considerably although not completely removed, and the distillates, which are similar in specific gravity to pure turpentine, would attain a tolerably high flash point, so that the danger of fire would be diminished. It would be supposed that varnishes diluted with these volatile distillates would dry readily on account of the rapid evaporation. But it has been found that when a varnish is diluted to the proper consistency, on the one hand, with turpentine, and, on the other, with benzine, the varnish containing turpentine sets and dries off much more rapidly than the varnish containing benzine, which even remains tacky for a long time. Since benzine and petroleum can absorb no oxygen from the air, whilst turpentine is capable of absorbing and also of transferring it, also since we know from chemical analysis that the oxidised compounds of linseed oil are not dissolved by benzine, an explanation has been found for the bad drying of varnish made from benzine.

Experiments, which were made with the object of preparing cheap terebine by using volatile photogene in place of turpentine, gave a negative result. The odour of this shale distillate was indeed not too strong, and could be covered by the addition of an essential oil of good perfume, but it was soon found that not only was the terebine deficient in drying power, but that a much larger " foot " formed than when turpentine was used.

Thus it appears that in the preparation of good terebines from linseed oil lead plaster, the use of any other solvent is excluded, and only the best turpentine should be used. Also, so-called Russian, Polish, German, and Finnish turpentines should not be used; these substances contain more or less resinous matter, and have not exactly the same chemical composition as other varieties of turpentine. They are obtained, together with tar, by distilling pine roots (*Pinus sylvestris*); these roots are very rich in resin, but they produce turpentine of small value, the penetrating smell of which adheres to other substances, so that it is not suitable for the preparation of good terebines. Even the turpentines from the same country, *e.g.* Finland, have all not the same composition, as Aschan and Hjelt have shown; one sample contained pinene, sylvestrene, and dipentene, which latter constituent was not found in other varieties. A turpentine from North Finland, obtained from pine stems as a by-product in the manufacture of pitch and wood tar, gave on distillation a relatively small fraction between 160° and 170° C., whilst 32·2 per cent. came over between 170° and 174° C., and 21·5 per cent. between 174° and 178° C. Turpentine from South Finland gave 7·1 per cent. at 155° to 160° C., 32·2 per cent. at 160° to 165° C., 22·6 per cent. at 165° to 170° C., and 20·1 per cent. at 170° to 175° C.; these distillates naturally differ in chemical composition.

It is thus quite erroneous to regard all unadulterated varieties of turpentine as of equal value; it will be advisable to reject Russian, Polish, German, etc., turpentines for the preparation of terebine, and to use only that variety obtained by distillation with or without water from the resin which flows naturally from trees.

Only these terebines which are made from rosin compounds may be prepared from the lower qualities of turpentine, since in this case only a cheap substance is required, of the activity of which large demands are not generally made.

The following directions may be given for the manufacture of terebines in general:—The linseed oil and powdered driers are placed in the pan and the heating commenced, whilst the materials are stirred from the beginning without interruption, so that they cannot settle and burn upon the bottom of the pan. With sufficiently intense heat the liquid begins to evolve bubbles in a relatively short time, and then becomes somewhat thicker; very soon afterwards a change in colour occurs, and the mixture froths up, when the pan must be taken off the fire or the latter be removed. The frothing and thickening of the mass take place very rapidly; the stirrer must be used on the surface so that the mixture does not come over. In the meanwhile the colour has darkened; when red lead is used it changes from red to a dark bronze brown, with other driers, to yellowish white or pale brown. A test is taken out and allowed to cool upon metal or glass; when cold it should be hard and breakable, it should not be soft or sticky; if the latter is the case the heating is continued until the necessary hardness is attained.

With a good fire the combination of the linseed oil with the driers takes place very rapidly, even when relatively large quantities of the latter are used; with a slow fire the process lasts longer and is less violent, on which account the slow process is preferred when large pans are used. Terebine may also be made in the steam-jacketed pan described in the chapter on boiled oil, but it will not be paler than terebine made over the fire with the same quantities of driers.

Formulæ for terebines—

(1)	7	kilos. of	tanked linseed oil.
	2	,,	red lead.
	2	,,	ground litharge.
	1	,,	sugar of lead.
(2)	7	kilos. of	tanked linseed oil.
	2	,,	shale brown.
	2	,,	pyrolusite.
	1	,,	red lead.
(3)	7	kilos. of	tanked linseed oil.
	2	,,	dehydrated sugar of lead.
	2	,,	red lead.
(4)	7	kilos. of	tanked linseed oil.
	1	,,	umber.
	1	,,	shale brown.
	1	,,	pyrolusite.
	1	,,	dehydrated sugar of lead.

(5) 7 kilos. of tanked linseed oil.
3 „ pure white lead.
1½ „ fused sugar of lead.
(6) 7 kilos. of tanked linseed oil.
2 „ manganous borate.
2 „ dehydrated zinc sulphate.
(7) 7 kilos. of tanked linseed oil.
2 „ manganous borate.
1½ „ dehydrated sugar of lead.

The process is the same with all these mixtures; when the boiled mass has somewhat cooled, 14 to 17 kilos. of turpentine are added whilst stirring. The terebine is then filtered through coarse cloth and tanked, so that it may brighten completely. The consistency, when the above-given quantity of turpentine is used, depends partially upon the strength, *i.e.* hardness, of the linseed oil plaster, and partly upon its temperature when the turpentine is added. If the mass is very hot more turpentine is volatilised than when it is cooler; the consistency of the finished substance varies accordingly.

Varnish extract.—A thick brown substance made exactly according to the preceding directions from

7 kilos. tanked linseed oil.
2 „ ground litharge.
2 „ red lead, the whole finally diluted with
4 „ boiled linseed oil.

English "Patent Driers"—

9·555 kilos. white sugar of lead.
35·055 „ powdered litharge.
47·805 „ zinc sulphate.
216·750 „ Paris white.
216·750 „ barytes.
106·550 „ raw linseed oil.

The first three materials in very fine powder are placed in a vessel containing 18 litres of oil; the mixture is allowed to stand 1 to 2 days, then well mixed, and 4½ litres of oil added.

After standing 2 to 3 days the mixture is again well stirred, and 4½ litres of oil added. In 10 to 14 days the mixture is ready, it is then ground with the Paris white, etc.

A formula for "Patent Driers"—

	(I)	(II)	(III)
Barytes	102	102	127·5
Paris white	102	76·5	127·5
White sugar of lead	12·750	19·20	19·12
Powdered litharge	25·50	12·75	19·12
Zinc sulphate	25·50	9·55	12·75
Dry white lead	25·50	12·75	14·57
Boiled linseed oil	51·00	38·25	51·00

Nothing is said as to the method of preparation.

Terebine.—This name is applied to a liquid drier which is still thinner than the liquid siccatives[1]; it is, however, nothing but linseed oil lead and manganese plaster dissolved in much turpentine, with an addition of copal. It is prepared as follows:—In a suitable pan are melted 2 kilos. of Manilla copal, or 2 kilos. of amber (for dark terebine), or 2 kilos. of Sierra Leone copal (for pale terebine). Then 0·5 kilo. of linseed oil is added, and 6 kilos. of one of the above-mentioned solutions of linseed oil lead plaster in turpentine, finally 14 kilos. of turpentine are introduced with constant stirring. The colour of the product varies according to the colour of the lead plaster and of the fused copal. Good terebine is fluid like turpentine, clear and transparent; a layer should be completely hard and dry in 30 minutes at the longest.

A formula for Terebine—

108 litres raw linseed oil.
17·3 kilos. flake litharge.

Boil 12 hours, and when cold enough mix with the turpentine (quantity not stated).

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[1] See, however, note on pp. 284–5 as to use of the term "terebine" in this country.

Taflin's siccative.—E. O. Taflin of Stockholm prepares a siccative by dissolving manganese oleate in oleïc acid; the product may be added to substances used in painting, such as linseed oil and other drying oils. It may also be added to varnishes made from any resin, in order to modify their brittleness. If crude turpentine or rosin be added to the drier, then a suitable solvent, such as petroleum or alcohol, and a suitable dry material, such as infusorial earth or china clay, together with a metallic oxide, such as zinc white, or also sulphur, a mass is obtained which well protects from wet. When applied to a wet surface, the mass produces in 2 days a hard, shining coating; it may also be mixed with paints. The rosin may be replaced by asphaltum, to which caoutchouc may be added.

LINOLEÏC ACID COMPOUNDS OF THE DRIERS

The linoleates of lead and manganese have been used for some time as driers in oil boiling. Use has long been made unwittingly of the property of linseed oil to produce lead and manganese linolates when treated with metallic oxides. All boiled oil made by the old process with lead and manganese compounds, also terebines made from linseed oil with large quantities of lead, are nothing more than salts of linoleïc acid, dissolved respectively in linseed oil or in turpentine. The metallic linoleates are applied exactly in the same manner as the corresponding resinates; they dissolve in linseed oil, absorb oxygen from the air, and otherwise behave exactly like the resinates, so that to treat them in detail would merely be repetition. The linseed oil lead plaster, which forms the basis of liquid driers, is simply lead linoleate with large quantities of uncombined lead.

Preparation of lead linoleate.—100 parts by weight of linseed oil are saponified by heating in a large iron pan with 15 parts of 77 per cent. caustic soda and 250 parts of water. The soap solution is then heated until it is quite clear. In another pan 60 parts of sugar of lead are dissolved by heating in 200 parts of water; the linseed oil soap, preferably diluted with a further quantity of water, is then allowed to run in to the lead acetate solution. Lead linoleate separates; it is freed from the liquid, repeatedly washed with hot water, dried, and then powdered. Occasionally it is fused in a pan with the addition of a certain quantity of finely ground litharge.

Manganese linoleate is obtained by precipitating the linseed oil soap solution, made as in the preceding paragraph, by a hot solution of 60 parts of manganous chloride in 200 parts of water, and treating the precipitate in a similar manner to the lead linoleate.

Lead-manganese linoleate, which is much used as a drier, is a mixture of 3 parts of lead linoleate and 1 part of manganese linoleate, made by fusing the constituents in a pan to a uniform mass.

THE METALLIC RESINATES AS DRIERS FOR LINSEED OIL

Since the metallic resinates, in particular the manganese compounds, were introduced into the preparation of linseed oil for painting, about 15 years have elapsed; during this period their use has continually increased, and they have largely displaced the older driers—pyrolusite, litharge, red lead, and lead acetate—in the manufacture of boiled oil. When they were first used objections were raised; in the introduction of resinates, an essential adulteration of boiled oil was perceived. But in a short time a more correct comprehension of the matter prevailed; it was seen that boiled oil, which contained 1 to 2 per cent. of a rosin compound, could not be described as "adulterated with rosin." The ready solubility of these substances in linseed oil, even in the cold, their remarkable powers of transferring oxygen from the air to the oil, which are not even approximately reached by any other manganese or lead compound, and the small formation of foot, have caused them to obtain so speedy an introduction into the manufacture of boiled oil that to-day nearly all boiled oil contains resinates.

The only resinates employed for this purpose are those of lead and manganese, the resinates of other metals, such as copper, barium, and iron, cannot transfer oxygen to linseed oil, and thus are quite useless if they do not contain lead or manganese in addition to the other metal.

The percentage of metal, with which the rosin acids are able to combine, is, in manganous compounds, about 8·3 per cent. of manganese; in manganic compounds, about 5·5 per cent.; and in lead compounds, about 31·4 per cent. of lead; thus these compounds contain on the average the following proportions of metal:—

Manganous resinate .	7·7	per cent.	of manganese.
Manganic " .	5·3	"	"
Lead " .	24·0	"	of lead.

The proportion of metal varies on account of differences in the raw materials, and further complications arise through different methods of preparation, which make the value of a theoretical view of these entirely technical products doubtful in the extreme.

The metallic resinates are prepared by two different processes—(1) by fusing rosin with the corresponding metallic oxide, and (2) by precipitating the alkaline rosin solution with a solution of the corresponding metallic salt. According to Weger, in the products obtained by fusion (erroneously called fused siccatives) the combination of the rosin acids with the metallic oxide, or the saponification of the ester by the latter, generally does not take place so regularly as the neutralisation of caustic soda by hydrochloric acid.

The reaction may stop at a certain stage; in addition, decomposition occurs owing to the high temperature, and when manganese compounds are used, oxidation; finally, it is not known whether the manganese is present as manganous or manganic oxide, or in both states. When the resinates are precipitated in the wet way these hindrances disappear, but at the same time the possibility of the formation of basic salts arises, which may also occur with fused resinates. The drier is required to be completely soluble, and to contain the metal in chemical combination with the organic acid, and not partially suspended as oxide. The total quantity of mineral matter cannot be taken as a measure of the value, since the metallic constituents (PbO, Mn_2O_3, $MnCO_3$, CaO, $CaCO_3$, etc.) which remain suspended when a soluble drier is used, are not only completely valueless, since they cannot act at the low temperature at which the oil is treated, but form a foot and turbidity which directly impair the quality of the oil. Thus the value of the drier is indicated by the proportion of metallic oxide, chemically combined with the rosin acid, and soluble in linseed oil at a temperature not exceeding 120° C. If, on the one hand, it is not desirable that soluble driers should contain uncombined mineral matter, on the other hand, little uncombined acid should be present, since the presence of free acid must diminish the proportion of active drier in the preparation. Thus, when insoluble lead and manganese are absent, the higher the proportion of soluble metal the better is the drier, and the smaller quantity is required to make linseed oil dry.

Experiments made by Weger on both a small and large scale, with the products of different works, have shown that fused manganic resinate rarely contains more than 3·2 per cent. of soluble manganese, precipitated manganous resinate rarely more than 6 per cent., and 7 per cent. only in exceptional cases. The lead-manganese resinate, which is most extensively used, comes into commerce under various fantastic names, and of the most varying composition. The best proportion of lead to manganese appears to be 5 : 1. The preparations of the best manufacturers agree tolerably on this point, as on most others; they contain 8 to 9 per cent. of soluble lead, and 1·5 to 2 per cent. of soluble manganese. If an exceptionally high percentage of soluble lead or manganese be found, it will be accompanied by much insoluble metallic compound, which is quite absent from few samples. In this case the drier will not form a clear solution, and the percentage of soluble metal will be very little higher than the figures given, so that in practice the advantage is not great. In spite of this, preparations overloaded with insoluble lead or manganese come continually into the market, and indeed more frequently than those which contain too much free rosin. Insoluble oxides and free rosin are, however, not mutually exclusive in the fused driers; they may occur together, which indeed is generally the case. On the other hand, driers of this class, adulterated with rosin, should not occur; low qualities, which contain more free rosin than is necessary, are certainly found, but they can hardly be regarded as intentionally adulterated, since it is difficult to draw a strict limit. Also there would be no great profit to the manufacturer in adulterating resinates.

In the analysis of soluble driers anhydrous ether is used to dissolve the manganese compound, and chloroform to dissolve lead resinate; the latter is soluble in turpentine, but is soon separated again. The solubility of the resinates in ether or chloroform respectively is parallel with the solubility in linseed oil, with the single difference that it is complete in the cold; thus the matter insoluble in cold ether or chloroform is also insoluble in warm linseed oil, and is valueless or even harmful. In the analytical estimation of the soluble compounds the drier is incinerated; the organic matter is thus destroyed. There is no object in weighing the residue, since the resinates often contain sand, and lead would also be present partly as metal and partly as oxide. In regard to the separation of lead and manganese little need be said, the latter is estimated volumetrically by potassium permanganate. If considerable quantities of calcium are present in addition to lead and manganese, after removal of the lead, the manganese and calcium are estimated together gravimetrically as carbonates, the manganese then titrated, and lime found from the difference.

When the total percentage of lead and manganese has been determined, the insoluble lead and manganese are found by dissolving in ether or chloroform, filtering, washing, incinerating the filter, etc. The soluble manganese is found from the difference; the result is controlled by measuring an aliquot part of the ethereal solution, evaporating the solvent, incinerating, etc. The soluble lead can only be found by difference, since the chloroform cannot be completely evaporated from the rosin solution, it escapes only at a red heat, when the greater portion of the lead volatilises as lead chloride.

Preparation of fused manganese resinate.—In manufacturing this substance a large iron pan is used, provided if possible with a mechanical stirrer, and heated over a fire. The flames should play only upon the bottom of the pan, in order to avoid overheating and the consequent decomposition and danger of fire.

Proportions—

Rosin	100 kilos.
Manganous hydrate . .	10 „

The rosin is introduced first into the pan; when it has melted and attained a temperature of at least 150° C. the introduction of the manganous hydrate is commenced; this substance must be in fine powder. It is introduced gradually, and the temperature raised to 200° to 220° C.; when all the manganous hydrate has been added, the heating is continued until the froth has fallen and only a thin scum remains on the surface; a deposit of undissolved manganese compound should also not be present. The contents of the pan are drawn off into the barrels in which the resinate is to be despatched.

The manganous hydrate may be obtained from chemical works, or made according to the following method:—500 kilos. of manganous chloride solution of 14½° B. (manganous chloride dissolved in 4 parts of water) are heated to 80° C.; 180 kilos. of caustic soda liquor of 25° B. are also heated to 80° C., allowed to run into the manganese liquor, and the mixture heated to boiling. The precipitate is washed once with water, allowed to settle, pressed as closely as possible, and dried in thin layers in the air. It is finally ground to a very fine powder in a suitable mill.

Preparation of precipitated manganese resinate.—The process by which this substance is made is essentially simple, but if the product is to be completely soluble it demands great care, and in particular a proper choice of the raw materials, upon the nature of which much depends. Large sheet-iron vessels, heated by steam, with mechanical stirrers, are most suitable for the dissolving and precipitating processes; the precipitate is best treated in a filter-press, it must be dried in a heated room. The mass, which becomes friable when dry, is ground by a disintegrator completely surrounded by a wood or iron casing.

Proportions—

100	kilos.	rosin.
55	„	caustic soda solution of 25° B., diluted with
345	„	water.
50	„	crystallised manganous chloride dissolved in
200	„	water (giving a solution of 14½° B.).

The caustic soda solution is first boiled and the rosin then introduced in small quantities whilst stirring. The heating is continued until the yellow foam has disappeared from the surface of the liquid, when 600 kilos. of boiling water are added, so that the 100 kilos. of rosin are now contained in 1000 kilos. of water. Evaporated water should be replaced, so that at the end of the dissolving process 1000 kilos. of water are still present. Such a dilute solution is necessary for the success of the operation.

The hot manganous chloride solution, at 14½° B., prepared in another pan from 50 kilos. of manganous chloride and 200 kilos. of water, is then allowed to flow slowly into the rosin solution with continual stirring. The mass at once becomes thick and difficult to stir; it is again heated up to 100° C.; it then becomes thinner, and the manganous resinate separates from the liquid. The aqueous solution is drawn off, the precipitate washed at least twice by introducing fresh water and heating up to 100° C., and finally removed from the pan. It is then freed from water as far as possible under a press, dried, and ground to fine powder.

The water used in this process should give no precipitate with alum, and the manganous chloride should be free from iron. By a large number of experiments it was determined that it is necessary to use exact quantities and Greenbank 98 per cent. caustic soda (Greenbank Alkali Works, St. Helens), and also to use a considerable excess of manganous chloride, otherwise the solution of the product, although at first clear, will afterwards give a large deposit. The complete solubility of the resinate in turpentine and benzine depends upon retaining the proper proportions, and using English caustic soda.

Preparation of lead resinate.—(1) 100 parts of rosin are melted in a suitable pan. When quite fused 12 parts of litharge are gradually added in small quantities with continual stirring, which must be continued at a gentle heat until the litharge is completely dissolved; the pan is then allowed to cool. The finished product is somewhat darker than the original rosin.

(2) 100 parts of rosin are brought into a pan with 160 parts of water and 15 parts of 77 per cent. caustic soda. The mixture is boiled until the rosin has completely dissolved and a clear solution of rosin soap has formed. At the same time 100 parts of sugar of lead are dissolved in 420 parts of boiling water; to this solution the rosin soap is now added, when thick masses of lead resinate are precipitated, which are washed with water, filtered off, and finally dried.

Preparation of lead-manganese resinate.—This substance is obtained by melting 100 parts of rosin with 6 parts of litharge and 2 parts of manganic hydrate in a manner similar to that used in the preparation of lead resinate.

Drier produced by Fertiliser Works.—This drier, the activity of which may be increased by adding a manganese salt (about 10 per cent. of pure manganous borate), is obtained by precipitating 70 parts of superphosphate of lime and 20 parts of superphosphate of magnesia with soda. The precipitate, which is an intimate mixture of the phosphates of lime and magnesia, is extremely fine, and when dried at a moderate temperature forms a pale hygroscopic powder which exerts considerable drying power when added to linseed oil.

CHAPTER IX

THE ADULTERATION AND EXAMINATION OF THE DRYING OILS AND BOILED OIL

ALL the drying oils and the substances made from them—boiled oil, printers' varnish, and liquid driers—are frequently subjected to adulterations of various kinds, which is the more safely done since these substances come into the hands of customers who are not in a position themselves to test them by simple methods, or to have them tested in a laboratory, since the cost would be high in proportion to the advantage gained. There is another reason, that chemists not specially skilled in the examination of oils would, in certain cases, have difficulty in determining whether a substance contained foreign additions.

The adulterants of the drying oils have been mentioned in Chapter II. in describing the individual oils, and it may here be stated that adulteration occurs much more rarely in wholesale dealings than would be supposed. On the other hand, products which have passed through three or four hands are far more frequently adulterated than is generally imagined. This is quite natural when we consider that an examination for adulteration is much less likely to be made, since it is certainly true that the small consumer possesses too little knowledge of the materials in question.

The following articles are adulterated:—*Drying oils.*—Linseed oil with non-drying oils (when the price admits), rosin, rosin oil and mineral oils (especially debloomed). Poppy oil and walnut oil with bleached linseed oil, but not with other fatty oils or with mineral oil. Hempseed oil is not exposed to adulteration, neither are sunflower oil and grape-seed oil , on the other hand, Chinese wood oil is adulterated with other native oils of less value.

Boiled oil with rosin, rosin oil, solutions of rosin soaps in benzine or petroleum, and with mineral oils, especially with debloomed oils and blown thick mineral oil.

Printers' varnish with rosin, rosin oil, and thickened blown mineral oil.

The drying oils are examined in regard to physical constants, which may be regarded as characteristic, specific gravity and boiling point, the estimation of the oxygen absorption when exposed in thin layers to air, and at the same time their property of drying to a solid skin. The reactions, of which there is a large number, proposed for detecting the various oils, especially when mixed with others, have been found in the course of time to be unreliable and to give contradictory results, because they depend on particular conditions. This is especially the case with the majority of the colour reactions produced by the action of sulphuric and nitric acids upon the different oils. Also the differences in the rise of temperature caused by the addition of strong sulphuric acid to the fatty oils are not sufficiently well defined. On the other hand, quantitative methods of examination have been successfully used in distinguishing the various oils ; to these belong the acid value, saponification value, ether value, Reichert-Meissl value, Hehner value, the acetyl, bromine, and iodine values, the importance of which is yet to be generally recognised.

The physical constants of those drying oils which are of technical value are given in the following tables :—

Candle-nut oil—

Specific gravity at 15° C. .	0·920–0·926 (de Negri).
Solidifying point . . .	– 18° C. (Oil extracted by petroleum ether remains fluid at this temperature.)

Hempseed oil—

Specific gravity at 15° C. . 0·9268 (hot pressed oil).
„ „ „ . . 0·9250 (cold pressed oil).
„ „ „ . . 0·925–0·9276.
Solidifying point: becomes thick at – 15° C.; solid at – 27°·5 C.

Chinese wood oil—

Specific gravity at 15° C. . 0·932–0·9413.
Solidifying point . . . not given.

Linseed oil—

Specific gravity at 15° C. . 0·9325–0·9347.
Boiling point . . . 320° C.
Solidifying point: according to Gusserow, the oil becomes solid after several days at – 16° C.; according to Chateau, – 27° C.; it melts at – 16° to – 20° C. (Glässner).

Poppy oil—

Specific gravity at 15° C. . 0·924–0·962.
Solidifying point . . . – 18° C.

Walnut oil—

Specific gravity at 15° C. . 0·9260 (hot pressed).
„ „ „ . 0·9250 (cold pressed).
Solidifying point: is still fluid at – 15° C., and begins to thicken at – 17° to – 18° C.; forms a transparent mass at – 24° C., and solidifies at – 27° to – 28° C. to a hard white fat.

Castor oil—

Specific gravity at 15° C. . 0·960–0·963.
Solidifying point . . . – 17° to – 18° C.
„ „ (American oil) – 10° to – 12° C.

Sunflowerseed oil—

Specific gravity at 15° C. . 0·924–0·926.
Solidifying point . . . – 17° C.

Grapeseed oil—

Specific gravity at 15° C. . 0·9202.
Solidifying point . . . – 12° C.

Deviations from these figures frequently occur, due to the nature of the seed, the manner in which the oil is obtained, and its age, so that they must always be regarded as only approximate for the different varieties of the same oil.

The specific gravity of the drying oils may be determined by means of the pyknometer, Sprengel's tube, or Westphal's hydrostatic balance. Sprengel's apparatus is a U-tube, the two arms of which terminate in capillary tubes bent at right angles at each side. The tube is completely filled with the oil, one end being dipped in the liquid, and the air sucked out from the other end. The tube is then brought into a water bath at a constant temperature; the oil which flows out is wiped away with filter paper. When the expansion of the oil ceases, the tube is taken out of the water bath, cooled, and weighed. The specific gravity may then be calculated, if the weight is known of the empty tube and of the tube filled with water.

Westphal's balance is shown in Fig. 42; the thermometer replaces a known volume of oil, so that the loss in weight is the weight of this volume of oil.

The chemical constants are:—

1. *The acid value*, which indicates the number of milligrammes of potassium hydroxide required to neutralise the free acid in the oil. It is determined by titrating the oil dissolved in alcohol by alcoholic or aqueous potash solution.

2. *The saponification value*, which was worked out by Köttstorfer, and is also known as Köttstorfer's value. It is determined as follows:—1·5 to 2 grms. of the oil are treated with 25 c.c. of semi-normal alcoholic potash on the water bath for 15 minutes. When the saponification is finished, 1 c.c. of alcoholic phenolphthaleïn solution is added, and the liquid titrated with semi-normal hydrochloric acid. A blank experiment is then made with 25 c.c. of alcoholic potash in

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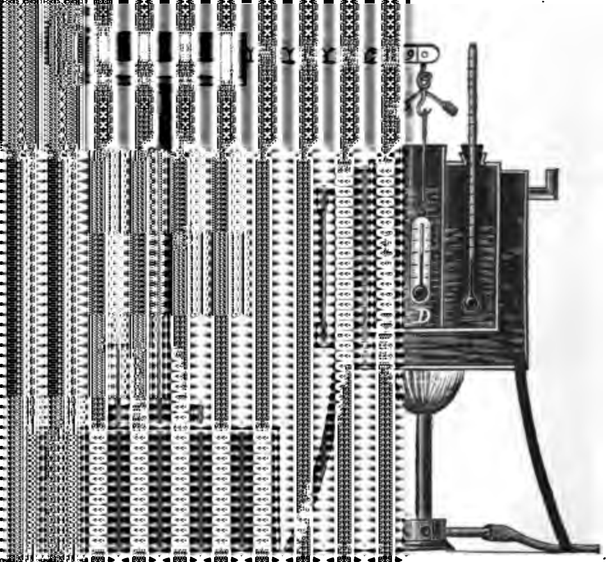

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oil are saponified with
of 70 per cent. alcohol

in a 200 c.c. flask upon the water bath. The alcohol is evaporated, the residue dissolved in 100 c.c. of water, and distilled with 40 c.c. of 10 per cent. sulphuric acid; 110 c.c. of the distillate are caught in a graduated flask, of which 100 c.c. are filtered into a second graduated flask, and titrated with decinormal alkali in the presence of litmus or phenolphthaleïn.

5. *The Hehner value* gives the percentage of insoluble fatty acids contained in the oil; 3 to 4 grms. of oil are saponified with 50 c.c. of alcohol and 1 to 2 grms. of caustic potash. After removal of the alcohol, the soap is dissolved in 100 to 150 c.c. of water, and sulphuric or hydrochloric acid added. The fatty acids are melted, and the mixture filtered through a paper dried at 100° C. The acids are washed with water until the filtrate is no longer acid.

6. *The acetyl value* gives the quantity of hydroxy-fatty acids or fatty alcohols contained in the oil. 20 to 50 grms. of the non-volatile fatty acids are acetylated by boiling for 2 hours with acetic anhydride. The product is several times boiled out with 500 to 600 c.c. of water, and the acids filtered in the air bath. The acetyl acid value is then determined by titration with aqueous semi-normal alkali, and the acetyl value by saponifying with alcoholic potash and titrating back with semi-normal hydrochloric acid.

7. *Bromine and iodine absorptions.*—The methods for estimating the bromine and iodine absorptions give the percentage of bromine or iodine taken up by the oil under conditions designed to cause the exclusive formation of addition products. The fatty acids of the acetic or stearic acid series are saturated substances which can form no addition products with bromine or iodine, whilst the acids of the acrylic or oleïc acid series can unite with 2 atoms of a halogen, and the acids of the propiolic or linolic series with 4 atoms of a halogen. The glycerides of the acids of these three series behave similarly to the free acids, so that the estimation

of the percentage absorption of iodine measures the proportion of oleïn to palmitin and stearin in a fat, and also the proportion of linolin in a drying oil, in comparison with the oleïn in a non-drying oil.

The estimation of the bromine addition does not give good results, whilst the estimation of the iodine value by Hübl's method gives constant results. Hübl uses an alcoholic solution of iodine and mercuric chloride; 25 grms. of iodine are dissolved in 500 c.c. of 95 per cent. alcohol free from fusel oil, also 30 grms. of mercuric chloride are dissolved in a second 500 c.c. of alcohol; the latter solution is filtered if necessary, and the two solutions then mixed. After standing 12 hours the solution is ready for use; its strength must be determined immediately before each experiment. The iodine value is determined as follows:—0·2 to 0·4 grm. of the oil is weighed off and dissolved in 10 c.c. of chloroform. Then 20 c.c. of iodine solution are added, and subsequently a further 5 to 10 c.c., so that the solution retains a dark brown colour after 2 hours; 10 to 15 c.c. of a 10 per cent. aqueous solution of potassium iodide and 150 c.c. of water are added, and the free iodine then titrated by sodium thiosulphate solution (24 grms. per litre). The absorbed iodine is then calculated into percentages of the oil, the result being termed the iodine or Hübl value. This value appears to be approximately constant for each oil or for each class; it is highest for the drying vegetable oils.

To facilitate reference, the chemical constants of the drying oils have been arranged in the following pages:—

Candle-nut oil—

Melting point of the fatty acids .	20°–21° C.
Solidifying ,, ,, . .	13° C.
Saponification value. . . .	187·4.
Iodine value	139·3.
Iodine value of the fatty acids . .	144·1.
Refractometer (Zeiss) at 15° C. .	76°–75°·5.

Hempseed oil—

Melting point of the fatty acids	19° C.
Solidifying „ „	15° C.
Saponification value	193·1.
Iodine value	143·0.
Iodine value of the free fatty acids	122–125·2.

Chinese wood oil—

Saponification value (Alkali Blue as indicator)	198·8, 199·5, 191·8, 191·0, 194.
Iodine value	107·6, 108·4, 156·1, 155·2, 165·7.
Acetyl acid value	187·6, 186·9, 173·5.
Acetyl saponification value	206·4, 204·1, 214·6.
Acetyl value	18·0, 41·1.
Refractometer	1°·80.
Melting point of the fatty acids	37° C.
Solidifying „ „	34° C.
Iodine value of the fatty acids	150·1.
Acid „ „ „	191·0.

Linseed oil—

Specific gravity of the fatty acids at 100° C.	0·8599.
Melting point of the fatty acids	11°·12 C., 17° C.
Solidifying „ „	13°·3 C.
Saponification value	189–195, 195·2.
Iodine value	158·0, 155·2.
Iodine value of the fatty acids	155·2–155·9.

Poppy oil—

Melting point of the fatty acids	20°·5 C.
Solidifying „ „	16°·5 C.
Hehner value	95·38.
Saponification value	194·6, 192·8.
Iodine value	136, 134.

Walnut oil—

Saponification value	196–197.
Iodine value	142–143.
Melting point of the fatty acids	20° C.
Solidifying „ „	16° C.

Castor oil—

Melting point of the fatty acids .	13° C.
Solidifying „ „ . .	3° C.
Saponification value. . . .	181–181·5.
Iodine value	84·4.
Iodine value of the fatty acids . .	86·6–88·3.

Sunflowerseed oil—

Saponification value. . . .	193·0.
Iodine value	129·0.
Hehner value	95·0.
Melting point of the fatty acids .	23° C.

Grapeseed oil—

No data are available.

The estimation of the iodine value of linseed oil leads to very varying results; without considering the older figures of Hübl and Moore, who obtained low results by using too small an excess of iodine, the values vary between 170 (Bendikt), 171 (Lewkowitsch), 172 (Holde), on the one hand, and 187 (Thomson and Ballantyne), 188 (Williams), and 195 (Archbutt), on the other hand. Without doubt, part of this difference is to be ascribed to estimations conducted in different ways. Thus the smaller figures are often too low, since the titre of the iodine solution has been taken after, instead of before, the experiment. Another cause of low results lies in allowing the reaction to proceed for too short a time, which is of great influence in the case of linseed oil, which adds iodine the most slowly of all oils, especially when the Hübl solution is not quite fresh. The differences are very great.

It appears from the researches of Thomson and Ballantyne and of Archbutt that differences between linseed oils of different source must be taken into consideration, as Lewkowitsch has again recently stated. Wijs, in order to measure this influence, has determined the iodine values of a number of oils of known source by the method, proposed by himself, with iodine chloride and glacial acetic acid. He finds a connection between iodine value and specific gravity, as is seen from the following collection of figures:—

Specific gravity of	1	oil of	iodine value	above 200	.	0·9352
Mean spec. gravity of	4	oils with	iodine values	195·1–200	.	0·9339
,, ,,	4	,,	,,	190·1–195·0	.	0·9329
,, ,,	4	,,	,,	185·1–190·0	.	0·9322
,, ,,	4	,,	,,	180·1–185	.	0·9317
,, ,,	4	,,	,,	below 180·1	.	0·9310

The specific gravity was taken at 15° C. with water at the same temperature as unity.

IODINE VALUES OF LINSEED OIL EXPRESSED FROM MIXED SEED

Mixture of Seed.	Iodine Value.
Dutch, Memel	199·1
Petersburg, Pernau, Königsberg	198·4
⅓ Viatka, ⅓ Petersburg, ⅓ Friesland	197·5
Viatka, Petersburg, Friesland	195·7
½ Petersburg, ½ Friesland	195·4
½ North Russia, ½ Friesland	195·1
⅔ Azov, ⅓ Dorpat	185·5
9/10 La Plata, 1/10 Azov	183·7
½ Azov, ¼ La Plata, ⅛ Calcutta, ⅛ North American	183·3
½ La Plata, ⅓ Bombay, ⅙ Taganrog	183·2
½ Azov, ⅜ Bombay, ⅛ La Plata	182·9
⅔ Bombay, ⅓ Taganrog	182·7
Bombay, Azov	181·5
South Russian, British Indian, North American	181·1
½ Bombay, ½ Azov	180·9
⅔ La Plata, ⅙ Bombay, ⅙ Calcutta	180·1
La Plata, Azov, Bombay	178·7

IODINE VALUES OF LINSEED OIL OF DIFFERENT SOURCES (WIJS)

Source of the Seed.	Iodine Value.	Specific Gravity.	Impurities. per cent.	Foreign Oleaginous Seed per cent.
Holland—				
Friesland	201·8	0·9352	...	...
,, ...	198·3	0·9346	...	...
,, ...	195·6	0·9333	...	...
,, ...	195·6	0·9337	...	...
,, ...	195·3	0·9339	...	...
Groningen	199·3	...	...	...
,, ...	197·4	...	...	...
Zeeland	193·5	...	...	...
British India—				
Bombay	187·5	0·9324	...	...
,, ...	186·0	...	4	...
,, ...	185·7	0·9320	...	...
,, ...	185·6	...	4·44	0·30
,, ...	185·0	...	6·55	0·59
,, ...	184·7	...	4·86	0·42
,, ...	183·9	0·9324	4·04	0·28
,, ...	182·3	0·9313	...	...
Calcutta	182·8	...	4	...
,, ...	182·2	0·9321	4·14	0·94
America—				
La Plata	182·7	0·9314	3·01	0·17
,, ...	180·0	...	2·17	0·00
,, ...	180·0	...	3·03	...
,, ...	179·6	...	...	...
,, ...	178·2	...	...	...
,, ...	177·5	0·9311	...	...
,, ...	176·3	0·9311	1·78	0·00
,, ...	174·7	...	4	...
North America	188·5	...	...	...
,, ...	182·3	...	2·37	...
,, ...	178·1	...	5·75	...
,, ...	178·1	0·9309	5	...
,, ...	177·8	...	...	...

IODINE VALUES OF LINSEED OIL—*continued.*

Source of the Seed.	Iodine Value.	Specific Gravity.	Impurities. per cent.	Foreign Oleaginous Seed per cent.
North Russia—				
Archangel	192·1	...	...	...
Viatka	197·4	...	...	...
,,	196·4	...	5	...
,,	194·0	...	...	...
Petersburg	195·0	0·9327	...	...
,,	194·2	...	...	...
,,	188·5	0·9325	...	...
Revel	198·5	...	8	...
Pernau	198·2	...	10	...
,,	196·9	...	5	...
Riga	200·0	...	5	...
,,	195·5	...	20	...
,,	194·2	...	...	...
Libau	195·5	...	5	...
,,	194·6	...	2	...
,,	192·4	0·9335	...	...
Not stated	194·6	0·9325	...	...
Mid-Russia—				
Samara	189·1	...	...	...
Steppes	188·9	...	2	...
South Russia—				
Azov	182·5	0·9319	...	...
,,	182·1	0·9312	...	...
,,	181·7	...	...	...
,,	179·9	...	...	...
,,	179·1	0·9314	..	...
,,	178·5	...	4	...
Taganrog	181·7	...	...	...
,,	178·3	...	...	...
,,	177·9	...	8·66	1·29
,,	176·3	0·9305	...	...
Not stated	186·3	...	...	...
,,	185·3	0·9318	...	...
,,	183·8	...	...	...
,,	182·8	...	...	...

The constants obtained for genuine samples of American linseed oil are given below .The ordinary methods were used. In the Maumené test the linseed oil was mixed with heavy mineral oil and the mixture left at rest for 1 to 2 hours before treatment with sulphuric acid; account was taken of the rise in temperature due to the mineral oil. The following table gives the figures obtained:—

Variety of oil.	Specific Gravity at 15° C.	Valenta Test. ° C.	Maumené Test. ° C.	Iodine Value		Drying Test. Hard after Hours.
				After 4 Hours' Action.	After 18 Hours' Action.	
Western States, raw .	0·933	79	97	174·7	180	72
,, ,, .	0·932	70	90	169·7	180	72
,, special	0·934	73	105	178·0	178	72
Old Calcutta . .	0·931	71·5	106	167·5	178	72
Eastern States . .	0·931	73	105	168·0	168	72
Western States, boiled .	0·936	74	100	178·8	178·8	18
Eastern States, boiled .	0·938	59·5	101	169·5	171	18
Bleached by acid . .	0·934	52·5	103	160·0	160	84
Bleached without acid .	0·932	60	105	157·0	162	84
Menhaden (fish) oil .	0·934	73·5	135	157·0	181(?)	84

The figures for Menhaden oil were added because it is used as an adulterant. In mixtures it lowers the Maumené test and the iodine value (?), and makes the drying slower. In such mixtures it will probably be found that the Maumené and iodine values are lower than the normal values, and that the iodine value shows an appreciable increase when the action is continued more than 4 hours.

The examination of raw and boiled linseed oil by means of the refractometer.—Weger has examined exhaustively the attempts made by Hefelmann and Mann, recently extended by Schick, to introduce the refractometer in the examination of boiled oil. Weger has tested the reliability of the process, and has unfortunately come to the negative conclusion that safe results cannot be attained in the examination of raw and boiled linseed oils, which was his original opinion, and that the process entirely fails when mineral oils are present. The refractometer gives a constant which at the most is of equal value with other constants — saponification value, amount of unsaponifiable matter; the instrument is, however, not adapted to a preliminary examination.

It was first found that all oils, which had been heated for a long time or had absorbed oxygen, gave a considerably greater deviation than the original raw oil. Long heating at a low temperature had the same effect as short heating to a high temperature, and spontaneous oxidation by standing in the air produced the same result as blowing in a current of air. By both manipulations, which cause both polymerisation and absorption of oxygen, the iodine value, and generally also the oxygen value, are diminished. On the other hand, the former belief that the refraction increased with the iodine value was shown not to be generally true, and at the most to be applicable to fresh raw oils, whilst the relation is the reverse for heated and oxidised oils, and thus for boiled oil. This result agrees with the observations of Späth on rancid fats. However, one of the samples behaved in the contrary way. It was further found that a differentiation of boiled oil, blown oil, and oil which had stood a long time in the air, was as little possible by means of the refractometer as the recognition of additions of rosin, rosin oil, or rosin preparations. The latter substances increase the refraction in the same way as the operations just mentioned. Similar results were obtained with the non-drying oils; in this case, too, thickening by oxygen absorption or polymerisation increases the refractive power.

REFRACTOMETER VALUES FOR RAW AND BOILED LINSEED OIL (WEGER)

	Refraction at 25° C.	Refraction at 40° C.	Mean Oxygen Absorption (layer process), per cent.
Raw Oils—			
Varnish oil	81·5	72·5	...
Artists' oil A	81·3	72·4	18·0
From Indian seed	80·2	71·4	17·1
Kept 3 years, not absolutely air tight	82·2	...	15·4
Of English origin, kept 5 years well corked	85·1	76·1	19·8
Artists' oil N	80·8	...	...
Treated Oils—			
Commercial sun-bleached	81·5	...	...
Artists' oil, 11 months exposed to air	83·0	...	...
Artists' oil, treated with Fuller's earth at 80° C.	81·5	72·5	...
Heated by superheated steam to 250° C.	81·5	72·5	15·3
Varnish oil, heated for some minutes at 180° C.	...	72·5	...
Artists' oil, heated for 6 hours at 150° C.	...	73·7	...
Artists' oil, freed from mucilage at 280° C.	81·0	...	...
Artists' oil, freed from mucilage at 280° C.	82·1	...	16·5
Artists' oil, freed from mucilage at 315° C.	82·5	...	...
Artists' oil, heated 40 hours at 180°–190° C.	90·9	...	...
Artists' oil, heated 5 minutes at 360° C.	102·3	...	5·4 (?)
Blown in mass at 100°–140° C., and kept 3 years, air not absolutely excluded	84·2	...	14·5
Blown in the cold, 3 years old	83·4	...	...
Artists' oil blown in the cold	...	73·6	...
Artists' oil blown hot	...	74·2	...
From Indian seed, blown	...	73·6	...
From Indian seed, blown 20 hours at 150° C.	92·6	...	...
From Indian seed, blown 25 hours at 150° C.	95·0	...	8·7
Commercial stand oil	99·3	...	11·1
Commercial thickened oil	92·9	...	10·7

In regard to the oils boiled with driers, the addition of 1 per cent. of a resinate increases the refraction by about 1°; the same effect is produced by the action of air or by heating for 1 hour to 150° C. On long keeping, the refraction of all boiled oil kept from contact with air does not further change, whatever was the method of preparation; on the other hand, when the bottles are open the oxygen absorption causes in all cases a considerable increase in the refraction. The influence of light, on the contrary, is small, especially when air is excluded, whilst when air can enter light appears to favour the oxygen absorption; but in this case the formation of a skin on the surface causes the results to be irregular. The refractions of different mineral oils, previously studied by Shick, vary within very wide limits, whilst rosin and rosin oil give very strong deviations, considerably above 105°, which, however, is also the case with Chinese wood oil. The refraction of turpentine alters rapidly and considerably on standing in the air. In general it follows from the figures given that the presence or absence of mineral oil cannot be ascertained from the refraction; indeed, a mineral oil of low refractive power might be used to hide a fraudulent addition of rosin or rosin oil; such additions may be readily discovered by other methods.

When the absence of mineral oil has been shown by other methods, it may indeed be said that boiled oil is unadulterated if its refraction falls within the limits of Hefelmann's figures. On the other hand, a higher refraction does not always indicate sophistication, although *most* commercial boiled oils have refractions within these limits, and *most* boiled oils containing mineral oil have greater refractive powers. The difference in the deviations of linseed oil at 25° and 40° C. amounts, as a rule, to 8·8 to 9 units, yet larger differences may be obtained.

Adulteration of boiled oil with rosin oil.—Especially since rosin oil has been made to dry, it has been the adulterant of boiled oil most frequently employed. It can generally be detected by the smell alone, which is more clearly perceptible when several drops of the boiled oil are warmed by rubbing between the hands. The taste also affords a means of detecting rosin oil; when present only in small quantity it is recognised by the harsh nauseating flavour. If dilute sulphuric or hydrochloric acid be mixed with boiled oil containing rosin oil, and the mixture left at rest, white sticky lumps are formed in addition to the lead or manganese precipitates, whilst pure boiled oil yields the precipitate alone, and becomes completely clear again after a few hours. The oil balance may also be used to test the oil; pure manganese boiled oil indicates 26°, lead boiled oil 24°, but oil containing rosin oil, barely 20° to 22° A characteristic property of all boiled oil adulterated with rosin oil is that, on standing in the cold, a portion of the rosin oil is separated; the oil becomes turbid and forms a compact foot, which completely disappears on warming. If suspected boiled oil be immersed in a freezing mixture at about —5° to —6° C. this appearance is observed in a short time in the presence of rosin oil, whilst pure boiled linseed oil remains bright and clear. If a considerable quantity of deposit forms in boiled oil on keeping, the oil is certainly adulterated with rosin oil. Rosin oil is detected by heating 10 grms. of the boiled oil with 50 c.c. of alcohol and 5 grms. of caustic potash, dissolved in the least quantity of water, under a reflux condenser for 30 minutes; 50 c.c. of water are then added, and the flask cooled by immersion in cold water. The liquid is shaken with petroleum ether in a separating funnel, and, when the two layers have separated, the lower is drawn off as completely as possible. The petroleum ether layer is washed repeatedly with water, which is not added to the original aqueous solution; finally the water is run off as completely as possible. In spite of the greatest care in running off the petroleum ether, drops of water accompany it; it is therefore not transferred directly to the weighed flask in which the ether is to be distilled off, but is brought into another dry flask. When it is poured from this into the weighed flask the water adheres to the sides of the first. The liquid, which has been once extracted, is extracted in a similar manner with petroleum ether to exhaustion, and the solvent is transferred to the weighed flask in the manner described. To decide rapidly whether the unsaponifiable matter obtained from the boiled oil by this process is rosin oil or mineral oil, it is shaken with an equal volume of acetone. If the two liquids mix completely, the oil is rosin oil, or a mixture of rosin oil with little mineral oil. Alcohol (specific gravity, 0·95) may also be used to decide; rosin oil sinks in this liquid, mineral oils do not.

Adulteration of boiled linseed oil with rosin.—Boiled linseed oil adulterated with rosin is generally more viscous than it should be; rosin is a solid substance, and its solution in linseed oil is naturally more viscous than the original oil. Such boiled oil dries in 36 to 48 hours, and is then apparently hard, but becomes sticky by the heat of the hand when the coating is touched. Dust sticks to the paint and makes it grey and unsightly; by a short exposure to the action of the atmosphere it is destroyed and falls off. If boiled oil, in which adulteration with rosin is suspected, be shaken with a weighed quantity of 95 per cent. alcohol at frequent intervals during several hours, the mixture left at rest, and the extract decanted off, the adulterant may be detected in it. The alcohol is distilled off in a retort or a flask provided with condenser; the residue is then weighed. If it amounts to more than $\frac{1}{40}$ of the extract, the oil is adulterated with rosin or rosin oil (according as the residue is solid or liquid), since raw or boiled linseed oil requires 40 parts of alcohol to dissolve it.

If boiled oil adulterated with rosin be exposed in a shallow vessel to the action of the air, a portion of the rosin separates in 10 to 14 days in the form of small crystalline grains which can easily be recognised as rosin.

The presence of rosin is detected in a somewhat more complicated manner by boiling the oil for several minutes with 95 per cent. alcohol, separating the solution after cooling, and adding a solution of lead acetate in water. If the boiled oil was pure merely a turbidity appears, but if rosin is present a flocculent white precipitant forms, which can be converted into pure rosin by washing repeatedly and melting.

Detection of rosin and rosin oil in fatty oils and boiled linseed oil.—According to Ulzer, the reaction of Liebermann—production of a fine red colour on the addition of strong sulphuric acid to solutions of resin acids in acetic anhydride—was first used by Storch to detect rosin oil, which gives a similar coloration with strong sulphuric acid when dissolved in acetic anhydride.

Later, Morawski successfully used this excellent reaction to detect rosin in the sizing of paper and in soaps, with the alteration that sulphuric acid of specific gravity 1·53 was used in the place of strong acid. He also studied the behaviour of the fatty oils when treated with acetic anhydride and sulphuric acid, and found that rosin and rosin oil may generally be detected by first shaking the oil with acetic anhydride, to which sulphuric acid of density 1·53 was then added. With some practice rosin and rosin oil may be detected with certainty by this process in most fatty oils.

Since it would be of great value to be able to use this simple reaction in the examination of boiled linseed oil (and varnishes), experiments were performed which give the following results:—Pure boiled linseed oil, obtained by boiling the oil either with litharge, red lead, manganese dioxide, or manganese borate, gives, when dissolved directly in very small quantities of acetic anhydride, a dark brownish-red coloration on the addition of sulphuric acid. Success was not attained by shaking the boiled oil with acetic anhydride, according to Morawski's method, because boiled oil is very soluble in acetic anhydride, and also emulsions readily form. In this special case it is more convenient to shake the boiled oil with absolute alcohol, allow to stand for several hours, evaporate the alcoholic solution, dissolve a portion of the residue in acetic anhydride, and add sulphuric acid. By this means a red coloration is obtained, but large quantities of rosin or rosin oil may be recognised by the reaction.

Adulteration of boiled linseed oil with fish oil rarely occurs, on account of the smell and taste of the latter, which are easily recognised. Linseed oil, adulterated with relatively small quantities of fish oil, has the characteristic smell and taste of the latter, and the adulteration is at once detected.

If 10 parts of the suspected linseed oil be stirred in a suitable vessel with 3 parts of sulphuric acid, and the mixture then left at rest until the oil and the acid layers have separated, a white precipitate is formed which contains the metallic compound; the oil itself is dark brown in the presence of fish oil, whilst the acid is orange-yellow or yellowish-brown. Pure boiled linseed oil, on the other hand, is at first greenish, and later brownish-green, whilst the acid acquires a more yellow colour. If adulterated boiled oil be treated with chlorine gas, it becomes at once dark brown and finally black, whilst unadulterated boiled oil becomes more and more colourless, since chlorine bleaches the vegetable fats, but turns all the animal fats, with the single exception of neatsfoot oil, steadily darker and finally black. If 5 volumes of boiled oil are boiled with 1 volume of caustic soda solution of 1·34 specific gravity, unadulterated oils produce a yellowish, oils containing fish oil a red, emulsion.

Distinction between boiled and unboiled linseed oil.—Linseed oil which has been made active by means of metallic compounds always contains a certain quantity of the metal in solution, which may be detected by heating the oil with acetic or dilute nitric acid and testing the separated aqueous liquid; the oil is thus recognised as boiled oil. The same result is given by the "boiled oils" made by means of driers without the application of heat; the method simply detects the presence of driers in linseed oil. The drying oils which have been treated only with atmospheric oxygen in a suitable apparatus, and which now frequently come into the market, contain no trace of metallic oxides, and thus cannot be easily recognised as boiled oil. Morpurgo has found that soaps made from unboiled, *i.e.* not heated, oils are completely separated from their aqueous solutions by the addition of common salt, whilst the soaps obtained from boiled linseed oil are incompletely separated even by excess of common salt. A portion of the soap remains in solution, as is also the case with rosin soap. The following process is recommended as giving good results:—20 grms. of the linseed oil under examination are saponified by an excess of caustic soda, common salt is then gradually added to the clear aqueous solution until soap no longer separates. The mixture is allowed to stand, and when the soap has collected the mixture is filtered. The clear filtrate is acidified strongly with acetic acid; if derived from a boiled oil, it becomes very turbid, if from unboiled oil it remains clear or nearly clear.

Lippert has made a number of experiments in regard to the oxygen absorption, *i.e.* increase in weight, of boiled oil mixed with mineral oils. He used the following mineral oils:—

American spindle oil, thin, specific gravity .	0·885
,, ,, viscous ,, .	0·903–0·909
Russian machinery oil, ,, ,, .	0·905–0·908
Pale yellow Zeitzer paraffin oil, very thin, specific gravity	0·890–0·900

The mineral oils themselves did not increase in weight during the experiment; the paraffin oil evaporated readily. Thus for them the oxygen absorption was zero. Lippert commenced his experiments with the conviction that, since mineral oils absorb no oxygen, or only when they contain asphaltum or resinous bodies, as the percentage of them in the drying oils increased, the maximum increase in weight of the mixture would be decreased in increasing degree. The maximum values attained by the oxygen absorption were as follow:—

Dutch linseed oil, 5 years old, mixed with American spindle oil, 0·903 to 0·909:—

	Per cent.
Linseed oil, pure, on 4th day	12·61
„ with 5 per cent. of spindle oil, on 4th day	12·13
„ „ 10 „ „ 6th „	11·38
„ „ 25 „ „ 6th „	9·65
„ „ 50 „ „ 7th „	6·03

Old boiled oil, made with manganic oxide, containing 0·4 per cent. of active manganese, mixed with Russian machinery oil, 0·905 to 0·908:—

	Per cent.
Boiled oil, pure, on 2nd day	11·93
„ with 5 per cent. of machinery oil, 2nd day	11·73
„ „ 10 „ „ 2nd „	11·34
„ „ 25 „ „ 2nd „	8·88
„ „ 50 „ „ 2nd „	4·76

Commercial boiled oil, made with manganic oxide, mixed with thin spindle oil, 0·885:—

	Per cent.
Boiled oil, pure, on 1st day	14·43
„ with 5 per cent. of spindle oil, on 1st day	13·40
„ „ 10 „ „ 1st „	13·35
„ „ 25 „ „ 1st „	10·03
„ „ 50 „ „ 3rd „	4·69

Equal parts of old manganic oxide boiled oil and litharge *boiled oil* (2·5 per cent. of PbO at 240° C.), mixed with thin spindle oil, 0·886 :—

	Per cent.
Manganic oxide oil, after 1 day	12·67
Litharge oil, after 16 hours	11·43
The mixed boiled oils—	
With 10 per cent. of spindle oil, after 16 hours	10·32
„ 25 „ „ „ 16 „	8·77
„ 50 „ „ „ 16 „	5·25

Varnish oil, mixed with volatile Zeitzer paraffin oil, 0·890 to 0·900 :—

	Per cent.
With 5 per cent. of paraffin oil, after 6 days	16·75
„ 10 „ „ „ 6 „	9·50
„ 25 „ „ „ 4 „	0·46
„ 50 „ „ (maximum decrease)	−13·15
Paraffin oil alone („)	−57·11

All the experiments were made by the "layer method,"—by observing the change in weight of thin layers spread upon glass plates. In the first place, they show (detailed figures are not given) that as the addition of mineral oil increases, the maximum increase in weight of raw and boiled linseed oil is decreased. In the case of the old Dutch linseed oil, the oxygen value decreased in exactly the same proportion as the addition of mineral oil increased. In general, adulteration with 10 per cent. of mineral oil would decrease the maximum increase to such an extent that adulteration would be suspected. But the case may occur that an adulterated oil gives a figure possible for a pure boiled oil, thus the commercial boiled oil made with manganic oxide attained a maximum increase of 13·35 per cent. in spite of the presence of 10 per cent. of spindle oil. The time of drying is not increased in every case by increasing quantities of mineral oil, but is never decreased, in particular the commencement of drying is never accelerated. In the case of the old manganese boiled oil, the commercial manganic oxide oil, and the mixture of manganese and litharge boiled oils, the time of drying is the same as of pure boiled oil, with the exception of the extreme adulteration with 50 per cent. of mineral oil, which particularly hinders the drying when the added oil is the spindle oil. In the experiments with old linseed oil and mineral oil of 0·9 specific gravity, increasing adulteration lengthens the time required to attain the maximum weight. It is natural that the properties of the mineral oil should have considerable effect. The results obtained with the varnish linseed oil are interesting, since a readily volatile adulterant was used; with the addition of 5 per cent. a high increase in weight was obtained, and the effect of the evaporation was not apparent. With an addition of 10 per cent. the oxygen value was considerably decreased, and with 25 per cent. the evaporation was almost greater than the absorption; after this point considerable decreases occurred. It is remarkable that both manganic oxide boiled oils when pure showed at first no oxygen absorption, *i.e.* the commencement of drying was delayed, the oil then suddenly attained the maximum weight; thus the initial stages are delayed, whilst when the lead boiled oil and mineral oil are added the absorption commences immediately. This cannot be due to a facilitation of the diffusion of oxygen by the mineral oil; the retardation is more probably due to the influence of moisture in the air, whilst the acceleration by the mineral oil of the commencement of drying is a consequence of movement taking place in the layer of oil. For it was found that shortly after the layers were spread out, and during the experiments, the mineral oils separated at the upper surface of the oil skin, so that after the maximum weight had been obtained the thin layer of mineral oil enabled the finger to slip over the oil skin, even when the adulteration was only 5 per cent., whilst the finger adhered to a pure oil skin. With the greater adulteration of 50 per cent. the whole of the mineral oil or of the volatile paraffin oil had collected in thick drops on the surface. This separation, which increases the usefulness of this method of testing oils, begins gradually with the oxygen absorption, but when much mineral oil is present it occurs in many cases immediately after the experiment is commenced, so that it is readily observed. The old sample of oil boiled with manganic oxide appeared to begin to dry more rapidly when mixed with 50 per cent. of mineral oil; in this case the separation of the mineral oil commenced at once, thus movement took place in the layer, and to this movement is to be ascribed, as we have said, the acceleration of the oxygen absorption in the case of the two manganic oxide boiled oils.

It might be imagined that mineral oils containing small quantities of drying oils would absorb oxygen, and that the thinner oils would do so rapidly if diffusion facilitated the oxygen absorption; it is yet uncertain whether adulterations of mineral oil with drying oils occur frequently in practice. In order to obtain results on this point, the spindle oils and the Russian mineral oil were mixed with 5 per cent. of linseed oil, which would attain alone an increase in weight of about 16 per cent.; but even after 3 days the thin spindle oil had absorbed only 0·5 per cent., whilst the two more viscous oils remained unaltered in weight. A slight increase was observed on the third day, which, however, might be due to other causes—deposition of dust.

Finally, it should be observed that the ease with which the oil is spread out and the appearance of fluorescence in raw and boiled linseed oil depend upon the nature and amount of the mineral oil added. These appearances are, however, of minor importance. The visible separation of mineral oil is an advantage characteristic of this process and gives it a practical importance.

The examination of oil paints (Loesner).—According to Loesner, the first requisite of a good oil paint is that it should form a homogeneous and non-porous layer on drying. The importance of this property is advanced because Simon alleges that the layers of most oil paints transmit water, as we have before mentioned. If sheet iron be painted, under certain circumstances rust forms after some time under the paint. If the iron be placed in water, no conclusion can be obtained as to the durability of the paint, because the action is much too slow; on the other hand, steam affords a very convenient method for testing the permeability of a layer of paint. Naturally, it is a matter of no importance whether large or small surfaces are tested, since a paint which cannot protect a small surface will not be able to prevent a larger one from rusting. The test is carried out in the following way: pieces of stout iron, 56 by 100 mm., are thoroughly cleaned on one side with sand or emery paper. The clean surface, which must not be touched with the fingers, is given two thin uniform coats of the paint, and dried for 4 days at the ordinary temperature. The greatest care must be taken that not the smallest portion remains unpainted. The paint is best dried by placing the sheets upon small strips of wood, in a horizontal position and the painted surface uppermost. On the back of the sheet may be marked the number of the paint under examination; this is best done with oil paint. The sheets are then laid over a boiling water bath, with the painted side downwards. In order to avoid mechanical injury, the short edges of the sheets are placed upon small rods of wood; the surface which comes in contact with these is to be disregarded. The height above the boiling water bath should uniformly be 50 mm., and the water should be kept at a constant level. After 15 hours the sheets are dried for a short time at 100° C. (not above). By means of a soft brush the layer of paint is saturated with aniline, so that it is softened and can readily be removed. It should be scratched off with a clean knife or another sharp instrument only when very hard, and then with great care, since the thin layer of rust which is formed under inferior paints is easily removed by the knife at the same time. When the paint has been removed the metal is dried by means of alcohol. In the case of a good paint, the surface should be completely intact; when this is the case, the scratches made in cleaning with sand paper are still present.

Many paints are praised as being fast to acids, by which the impression is created that they are of some special nature. The fastness to acids frequently depends only upon the property, more or less common to all oil paints, of being little changed by dilute sulphuric acid. In order to decide whether a paint is really fast to acids, the painted surface is placed over a glass vessel, containing 100 c.c., half filled with strong hydrochloric acid (33 per cent.), so that the layer of paint lies horizontally 50 mm. above the acid. Very soon the acid vapours condense on the paint, and in many cases form strong salt solutions from the oxides contained in the paint, which in their turn protect the iron from rusting. However, if the layer of salts be removed every hour by washing the plates, and if the plates be dried and again exposed to the action of the acid vapours, eventually warming the acid to 20° C., the destructive action of the hydrochloric acid is soon observed. If still apparently intact after 12 hours the coat of paint is removed by means of aniline in the above-mentioned manner. The action of the acid can then be seen clearly; it should be unnecessary to state that this action can only consist in etching and not in the formation of rust.

[illegible]

[illegible] weighed. The difference is the organic matter.	[illegible] residue weighed and further examined.	[illegible] metals, which were present as soaps.	[illegible] residue weighed. It contains the free fatty and resin acids of the original sample, and if aluminium or metallic soaps were present, the fatty acids resulting from their decomposition.	[illegible] fats. When the solution is treated with hydrochloric acid, shaken with ether, and the ethereal solution evaporated, the weight of the residue of fatty acids multiplied by 1·055 gives approximately the weight of the neutral oil.	[illegible] moved by a current of air. Hydrocarbons remain.

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